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FOREWORD

The *Proceedings* of the American Society for Testing and Materials are published annually and include all reports and papers offered to the Society during the current year and accepted by the Administrative Committee on Papers and Publications for the *Proceedings,* together with discussion.

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A list of the Special Technical Publications published by the Society in 1963 is given in this volume (see Contents). This supplements the lists appearing in the *Proceedings* from 1948 to 1962 covering all special publications published by the Society up to and including 1962. Also included is a list of papers appearing in *Materials Research & Standards.*

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Gillett Memorial Lecture

Trends in the Metallurgy of Low-Alloy, High-Yield-Strength Structural Steels—James B. Austin (*Issued as a separate publication*)

Lecture on Outstanding Research

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SUMMARY OF PROCEEDINGS

OF THE

SIXTY-SIXTH ANNUAL MEETING

ATLANTIC CITY, N. J., JUNE 23–28, 1963

This summary of the Sixty-sixth Annual Meeting of the American Society for Testing and Materials, held at the Chalfonte-Haddon Hall Hotel, Atlantic City, N. J., June 23–28, 1963, is a record of the transactions of the meeting, including the actions taken on the various recommendations submitted by the technical committees. In all, 39 technical sessions were held.

The registered attendance of the meeting is as follows: Members present or represented, 2002; committee members, 1184; guests, 581; total, 3767; ladies, 462.

The Proceedings are set forth session by session. There were 88 reports and 160 formal papers presented. The record with respect to each has been briefed; the recommendations in the reports have been grouped so as to cover the acceptance of material for publication as tentative, such as new specifications, methods of test, revisions of tentatives, and proposed revisions of existing standards, and, as a separate group, the approval of matters that were referred to letter ballot of the Society, comprising the adoption of tentatives as standard, and the adoption of revisions of standards. Accordingly, wherever the action is reported "adopted as standard" or "adopted as standard, revisions in," it is understood that this indicates approval of the Annual Meeting for reference to letter ballot of the Society.[1] The various recommendations so recorded are included in the Society letter ballot. The actions designated as "accepted as tentative" or "accepted as tentative, revisions in" are self-evident as indicating acceptance by the Society at the Annual Meeting for publication as tentative. Designations that have since been assigned to new tentatives are included as information.

All papers and committee reports, unless otherwise noted, will be published in the *Proceedings*.

While all the items on the program are recorded under the particular session in which they are presented, for convenience in locating actions with respect to any particular report, the accompanying list is presented of all reports together with the page reference where the actions thereon are recorded.

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[1] The letter ballot on recommendations affecting standards, distributed to the Society membership, will be canvassed on September 30, 1963. See Editor's Note, p. 44.

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SESSION ON FRACTURE TESTING OF METALS

SUNDAY, JUNE 23, 8:00 P.M.

SESSION CHAIRMAN: R. H. HEYER

An Apparatus for the Direct Determination of Ductile-Brittle Temperatures—J. H. Westbrook and D. L. Wood, presented by Mr. Wood.

Influence of Section Size on the Mechanical Properties and Fracture Toughness of 7075-T6 Aluminum and 6Al-6V-2Sn Titanium and AISI 4340 Steel—T. S. DeSisto, F. L. Larson, and F. L. Carr, presented by Mr. DeSisto.

Comparison of Various Test Specimens for the Determination of Notch Sensitivity and Fracture Toughness of High-Strength Sheet Materials—C. F. Hickey, Jr., and F. L. Larson, presented by Mr. Hickey.

Electrical Potential Technique for Determining Slow Crack Growth—A. A. Anctil, E. B. Kula, and E. DiCesare, presented by Mr. Anctil.

An Acoustic Method for Determining the Onset of Crack Propagation in Sharply Notched Specimens—M. H. Jones and W. F. Brown, Jr., presented by Mr. Brown.

Effects of Several Alloy Production and Fabrication Variables on the Sharp Notch Properties of 5.0Al-2.5Sn Titanium Sheet at Liquid Hydrogen Temperatures—J. L. Shannon and W. F. Brown, Jr., presented by Mr. Shannon.

SYMPOSIUM ON SOIL EXPLORATION[2]

(*Sponsored by Committee D-18 on Soils for Engineering Purposes*)

SUNDAY, JUNE 23, 8:00 P.M.

SESSION CHAIRMAN: A. I. JOHNSON

ICBM Site Selection and Subsurface Site Investigations (WS-133A Minuteman)—K. H. O'Brien, presented by the author.

Reconnaissance Exploration of Subsurface Conditions by Airphoto Interpretation—D. K. Erb, presented by M. D. Morris.

Research in the Engineering Geology of the Straight Creek Tunnel Site, Colorado—C. S. Robinson and F. T. Lee, presented by Mr. Robinson.

Calibration of Nuclear Meters for Measuring Subsurface Moisture and Density—W. F. Troxler, presented by the author.

(*Continued on Monday, June 24, at 2:00 p.m. and 8:00 p.m.*)

OPENING SESSION

MONDAY, JUNE 24, 9:00 A.M.

FORMAL OPENING OF THE SIXTY-SIXTH ANNUAL MEETING, PRESIDENT A. E. WEBBER

The session was formally opened by President A. E. Webber, who called attention to the very complete program of events that were to follow during the ensuing week. The meeting was then turned over to the session chairman, L. P. Spalding.

SYMPOSIUM ON MATERIALS PROBLEMS IN THE DESIGN OF A SUPERSONIC TRANSPORT AIRCRAFT[3]

(*Sponsored by Administrative Committee on Similated Service Testing*)

SESSION CHAIRMAN: L. P. SPALDING

The Supersonic Transport and the Materials Problems It Poses—F. R. Kostoch, presented by the author.

Materials for Wings and Fuselage of Supersonic Transports—R. H. Raring, presented by the author.

Nonmetallics for a Supersonic Transport—H. Hightchew, presented by the author.

Fuels and Lubricants for the Supersonic Transport—R. C. Sheard, presented by the author.

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[2] Issued as *ASTM STP 351*.

[3] Published in *Materials Research & Standards*, Vol. 3, No. 10, Oct., 1963, p. 805.

COMMITTEE REPORT SESSION

MONDAY, JUNE 24, 11:30 A.M.

SESSION CHAIRMAN: W. A. GLOGER

Committee A-9 on Ferro-Alloys:

Report presented by S. W. Poole, chairman, and accepted as a report of progress.

Committee B-3 on Corrosion of Nonferrous Metals and Alloys:

Report presented in the absence of the chairman by E. K. Camp, vice-chairman, and accepted as a report of progress.

Committee B-9 on Metal Powders and Metal Powder Products:

Report presented in the absence of the chairman by F. Y. Speight, ASTM staff, and accepted as a report of progress.

Committee C-6 on Pyrolytic Materials:

Report presented in the absence of the chairman by F. Y. Speight, ASTM staff, and accepted as a report of progress.

Committee D-7 on Wood:

Report presented by L. J. Markwardt, chairman, and the following actions taken:

Accepted as Tentative:

Test for Structural Insulating Roof Deck (D 2164 - 63 T)

Accepted as Tentative, Revisions in:

Methods of Evaluating the Properties of Wood-Base Fiber and Particle Panel Materials (D 1037 - 60 T)
Def. of Terms Relating to Wood-Base Fiber and Particle Panel Materials (D 1554 - 60 T)
Spec. for Pressure Treatment of Timber Products (D 1760 - 62 T)
Methods of Testing Metal Fasteners Employed in Wood Construction (D 1761 - 60 T)

Adopted as Standard:

Method for Accelerated Laboratory Test of Natural Decay Resistance of Woods (D 2017 - 62 T)
Test for Moisture and Creosote-Type Preservative in Wood (D 1860 - 61 T)
Spec. for Creosote - Coal Tar Solution (D 391 - 61 T)
Spec. for Creosote - Petroleum Solution (D 1858 - 61 T)
Spec. for Petroleum for Blending with Creosote (D 1859 - 61 T)

Adopted as Standard, Revisions in:

Methods of Testing Veneer, Plywood, and Other Glued Veneer Constructions (D 805 - 52)

Committee E-5 on Fire Tests of Materials and Construction:

Report presented in the absence of the chairman by I. A. Benjamin, secretary, and the following action taken:

Accepted as Tentative, Revision in:

Methods of Fire Tests of Building Construction and Materials (E 119 - 61)

Committee E-8 on Nomenclature and Definitions:

Report presented by F. W. Reinhart, chairman, and accepted as a report of progress.

Committee E-13 on Absorption Spectroscopy:

Report presented in the absence of the chairman by R. F. Robey, secretary, and the following actions taken:

Accepted as Tentative, Revisions in:

Def. of Terms and Symbols Relating to Absorption Spectroscopy (E 131 - 62 T)

Adopted as Standard:

Rec. Practices for General Techniques of Ultraviolet Quantitative Analysis (E 169 - 60 T)

Accepted for Publication as Information Only:

Rec. Practice for Describing and Measuring Performance of Spectrophotometers

Committee F-4 on Surgical Implant Materials:

Report presented by H. R. Lissner, chairman, and accepted as a report of progress.

Joint Committee on Leather:

Report presented by B. L. Lewis, vice-chairman, and the following actions taken:

Accepted as Tentative:

Test for Tensile Strength of Leather (D 2209 - 63 T)

Test for Grain Crack and Extension of Leather by the Mullen Test (D 2210 - 63 T)

Test for Elongation of Leather (D 2211 - 63 T)

Test for Breaking Strength of Leather by the Grab Method (D 2208 - 63 T)

Test for Bursting Strength of Leather by the Ball Method (D 2207 - 63 T)

Test for Slit Tear Resistance of Leather (D 2212 - 63 T)

Adopted as Standard:

Test for Resistance to Wetting of Garment-Type Leathers (Spray Test) (D 1913 - 61 T)

Accepted for Publication as Information Only:

Method of Measuring Resistance of Chrome-Tanned Shoe Upper Leather to Artificial Perspiration. (New evidence presented by a member of the joint committee subsequent to the Annual Meeting throws the method into serious question and the staff was requested to withhold publication of the method.)

SESSION ON METALS

MONDAY, JUNE 24, 2:00 P.M.

SESSION CHAIRMAN: I. V. WILLIAMS

The Thermal Expansion of Five Titanium Carbide Cermets from 68 to 1800 F—L. C. Harrington and G. H. Rowe, presented by Mr. Rowe.

The Thermal Expansion of Thirteen Tungsten Carbide Cermets from 68 to 1800 F—L. C. Harrington and G. H. Rowe, presented by Mr. Rowe.

Mechanical Properties of Cold-Drawn Martensitic SAE 4340 Steel Rod—E. DiCesare presented by the author.

Suppression of the Bauschinger Effect and Changes in Flow Pattern of Ductile Metals Caused by Cyclic Torsional Strains—N. H. Polakowski, presented by the author.

Mechanical Properties of a Chromium-1 per cent Columbium Alloy—N. J. Grant and R. E. Cairns, presented by Mr. Cairns.

SESSION ON GENERAL TESTING

MONDAY, JUNE 24, 2:00 P.M.

SESSION CHAIRMAN: H. N. BOGART

C-Notch Impact Test for Steels at High Hardnesses—G. Steven and J. R. Handyside, presented by Mr. Steven.

An Investigation of Subsize Charpy Impact Specimens—N. H. Fahey and E. B. Kula, presented by Mr. Fahey.

A Technique for Correcting Sieve Results to Nominal Values—E. R. Goffaux, presented by the author.

Studies in Contact Microradiography—R. W. McClung, presented by the author.

Tension Tests of Metals at Strain Rates up to 200 sec—J. E. Smith, presented by title only.[4]

SYMPOSIUM ON SOIL EXPLORATION[2] (*Cont.*)

MONDAY, JUNE 24, 2:00 P.M.

SESSION CHAIRMAN: J. P. GNAEDINGER

An Investigation of a Group of Surficial Soils by the Field Vane Test—T. K. Liu and T. H. Thornburn, presented by Mr. Liu.

Shear Strength Determination of Soft Clayey Soil by Field and Laboratory Methods—E. B. Hall, presented by the author.

Subsurface Exploration by Sonar Seismic

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[4] Published in *Materials Research & Standards*, Vol. 3, No. 9, Sept., 1963, p. 713.

System—E. D. Van Reenan, presented by the author.

Experience with Vane Probes in Alluvial and Eolian Deposits—H. M. Reitz, presented by the author.

Application of Geophysical Logs to Some Soil Engineering Problems—H. Guyod, presented by A. I. Johnson.

(*Continued at 8:00 p.m.*)

PANEL DISCUSSION ON STANDARDIZATION IN THE NUCLEAR FIELD

(*Sponsored by Special Administrative Committee on Nuclear Problems*)

MONDAY, JUNE 24, 2:30 P.M.

A Panel Discussion was held on Standardization Activities in the Nuclear Field sponsored by the Special Administrative Committee on Nuclear Problems. This consisted of individual reports of ASTM committee activities, standardization activities in The American Society of Mechanical Engineers and American Standards Assn., and various industry reports on the status of the art.

SYMPOSIUM ON SOIL EXPLORATION[2] (*Cont.*)

MONDAY, JUNE 24, 8:00 P.M.

SESSION CHAIRMAN: R. F. LEGGET

Research on Mechanical Methods of Undisturbed Soil Sampling—K. R. Clark, presented by the author.

Materials Exploration for Oroville Dam—A. L. O'Neill and R. G. Nutting, presented by Mr. Nutting.

NX Bore Hole Camera—John Trantina and Lloyd Cluff, presented by Mr. Trantina.

Research on the Detection and Interpretation of Microseisms in Soil Masses—C. P. Fisher and C. A. Yorke, presented by Mr. Fisher.

Reverse Circulation Drilling—An Improved Tool for Production Wells and Exploratory Holes—W. L. Burnham, presented by the author.

SYMPOSIUM ON ADVANCES IN THE TECHNOLOGY OF STAINLESS STEELS AND RELATED ALLOYS[2a]

(*Sponsored by Committee A-10 on Iron-Chromium, Iron-Chromium Nickel and Related Alloys*)

MONDAY, JUNE 24, 7:30 P.M.

CO-CHAIRMEN: R. B. GUINA
F. K. BLOOM

Special Processing and Fabrication

The Application of Electron-Beam and High-Frequency Resistance Welding to the Fabrication of Stainless Steel—H. W. Mishler, H. E. Pattee, J. J. Vagi, and D. C. Martin, presented by A. M. Hall.

Effects of Vacuum Melting and of Vacuum Annealing on the Properties of Austenitic Stainless Steels—D. B. Roach, W. B. Leffingwell, and A. M. Hall, presented by Mr. Hall.

Effects of Austrolling on the Properties of Crucible 422 Stainless Steel—R. C. Westgren and E. J. Dulis, presented by Mr. Westgren.

High-Strength Stainless Steel by Deformation at Low Temperatures—S. Floreen and J. R. Mihalisin, presented by Mr. Floreen.

The Mechanisms of Deformation and Work Hardening in AISI Type 301 Stainless Steel—W. F. Barclay, presented by the author.

Practical Aspects of Production Bright Annealing of Stainless Steel—R. C. Boyer and R. J. Perrine, presented by Mr. Boyer.

[2a] To be issued as a separate publication.

Plasma Arc Cutting and Welding Stainless Steel—R. L. O'Brien, presented by the author.

Advancements in Extrusion and Cold Finishing of Stainless Steel Irregular Shapes—J. J. Barrett, presented by title only.

(*Continued on Tuesday, June 25, at 9:00 a.m., 2:00 p.m., and 7:30 p.m.*)

SESSION ON CONCRETE

(*Sponsored by Committee C-9 on Concrete and Concrete Aggregates*)

MONDAY, JUNE 24, 8:00 P.M.

SESSION CHAIRMAN: F. J. MARDULIER

Volume Change as a Measure of Freezing and Thawing Resistance of Concrete—M. H. Wills, Jr., R. D. Gaynor, S. Walker, and H. A. Lepper, presented by Mr. Wills.

Behavior of Aluminous Cement Concretes at High Temperatures Incorporating Different Aggregates—N. G. Zoldners, V. M. Malhotra, and H. S. Wilson, presented by Mr. Zoldners.

Accelerated Strength Testing of Concrete Cylinders—P. Smith and B. Chojnacki, presented by Mr. Smith.

Effect of Sampling Procedure and Short-Term Job Curing on Measured Statistical Variation of Compressive Strength of Concrete—W. K. Wagner, presented by the author.[5]

The Dielectric Properties of Hardened Concrete with Respect to Their Utility as Moisture Indicators—J. R. Bell, G. A. Leonards, and W. L. Dolch, presented by Mr. Dolch.

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[5] Published in *Materials Research & Standards*, Vol. 3, No. 8, Aug., 1963, p. 629.

SYMPOSIUM ON ADVANCES IN THE TECHNOLOGY OF STAINLESS STEELS AND RELATED ALLOYS[2a] (*Cont.*)

TUESDAY, JUNE 25, 9:00 A.M.

CO-CHAIRMEN: J. STRAUSS
W. F. SIMMONS

Alloy Development: Part I

New Martensitic Age Hardening Stainless Steels—G. N. Aggen, C. M. Hammond, and R. A. Lula, presented by Mr. Aggen.

Mechanical Properties and Corrosion Resistance of a High-Strength Chromium-Manganese-Nitrogen Stainless Steel—J. J. Heger, presented by the author.

Metallurgy of a Columbium Hardened Nickel-Chromium-Iron Alloy—H. S. Eiselstein, presented by the author.

Properties of 12 per cent Chromium Alloys Modified with Small Columbium Additions—H. Tanczyn, presented by the author.

A Wrought High-Strength Weldable Stainless Steel for Elevated-Temperature Service—F. C. Hull, presented by the author.

(*Continued at 2:00 p.m. and 7:30 p.m.*)

SYMPOSIUM ON THE CHEMICAL AND PHYSICAL EFFECTS OF HIGH-ENERGY RADIATION ON INORGANIC SUBSTANCES[6]

(Sponsored by Committee E-10 on Radioisotopes and Radiation Effects)

TUESDAY, JUNE 25, 9:00 A.M.

SESSION CHAIRMAN: E. S. FREEMAN

The Use of Color Centers for the Detection and Measurement of Radiation-Induced Defects—P. W. Levy, presented by the author.

Comparison of Electron Paramagnetic Resonance Studies of Well-Defined Systems and Irradiated Substances—R. C. Sahni, presented by the author.

Thermal Decomposition of Irradiated Solids, Part I—E. G. Prout, M. E. Brown, and G. S. Woods, presented by Mr. Prout.

Radiation-Induced Changes in the Chemical Reactivity of Ammonium Perchlorate—E. S. Freeman and D. A. Anderson, presented by Mr. Freeman.

(Continued at 2:00 p.m.)

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[6] Issued as *ASTM STP 359.*

SESSION ON FATIGUE

(Sponsored by Committee E-9 on Fatigue)

TUESDAY, JUNE 25, 9:00 A.M.

SESSION CHAIRMAN: C. L. KENT

A Device for Low-Cost Fatigue Testing—V. D. Coombs, presented by the author.

Effects of Pin Interference and Bolt Torque on Fatigue Strength of Lug Joints—A. A. Mittenbergs and L. G. Beall, presented by Mr. Mittenbergs.

Application of Induction Heating to High-Temperature Fatigue Testing—D. L. Harper› W. H. H. Feilbach, and J. F. Libsch, presented by Mr. Libsch.

Improvement in Aluminum-Alloy Fatigue Life by Cold-Working—R. E. Whaley, presented by the author.

(Contiuned at 2:00 p.m.)

PRESIDENT'S LUNCHEON AND BUSINESS MEETING

TUESDAY, JUNE 25, 12 NOON

TOASTMASTER: PAST-PRESIDENT MILES N. CLAIR

INVOCATION: ARTHUR MCKAY ACKERSON, PASTOR, ALL SAINTS EPISCOPAL CHURCH, ATLANTIC CITY, N. J.

There were 240 present at the luncheon, including a number of ladies and visitors.

After his opening remarks, the toastmaster introduced the following guests who were seated at the head table: Dr. James B. Austin, 1963 H. W. Gillett Memorial Lecturer; Dr. Richard F. Blomquist, 1963 Edgar Marburg Lecturer; Mr. Charles L. Kent, senior vice-president of ASTM; Mr. Raymond E. Hess, associate executive secretary of ASTM; and Mr. Fred F. Van Atta, ASTM treasurer.

Election of Officers:

The results of the letter ballot on election of new officers were reported by Clyde B. Jenni on behalf of the Teller's Committee consisting of Mr. Jenni and Walter P. Heck. The results were as follows:

For President, to serve for one year:
  I. Vernon Williams, 2878 votes
For Vice-President, to serve for one year:
  Robert F. Legget, 2846 votes
For Directors, to serve for three years:
  Harold N. Bogart, 2816 votes
  Walter A. Gloger, 2821 votes
  William L. Glowacki, 2807 votes
  Harold M. Smith, 2832 votes
  Luther P. Spalding, 2826 votes
  Lyman W. Wood, 2845 votes

The newly elected directors were introduced with the exception of Messrs. Spalding and Wood who were not present. President-elect Williams and Vice-President-elect Legget were introduced in turn and responded briefly.

Recognition of Retiring Directors and Past-Presidents:

President Webber presented certificates of appreciation to the directors retiring from the Board. Present to receive their certificates were Arthur R. Belyea, Harry M. Hancock, Jerome J. Kanter, William R. Willets, and retiring Past-President Frank L. LaQue. Also retiring from the Board but unable to attend the luncheon was Jay C. Harris.

Recognition of Fifty- and Sixty-Year Members:

Past-President Clair read the names of four personal and 19 industrial or institutional members who had maintained continuous membership in ASTM for 50 years. Likewise, the names of four industrial and institutional organizations that had been members of ASTM for 60 years were read. These members or their representatives who were present were asked to rise. Certificates recognizing the achievement of both 50- and 60-year memberships were awarded at the close of the luncheon.

Conferral of Honorary Membership:

The executive secretary read citations for four members of the Society who had been elected to honorary membership by action of the Board of Directors on January 15, 1963: Robert Burns, George H. Harnden, Max Hecht, and Francis M. Howell. Certificates of honorary membership were presented to them by President Webber.

President's Address:

Alfred C. Webber delivered the annual president's address, entitled this year "ASTM—Our Heritage and Responsibilities."

BUSINESS MEETING OF THE SOCIETY

Under the chairmanship of President Webber, a brief business meeting was held. The president announced that the $6500 ASTM Doctoral Fellowship for the academic year 1964–1965 had been awarded to The Pennsylvania State University and that the University of California, Los Angeles, the school that had received the 1963–1964 ASTM Doctoral Fellowship, had selected James Kaae as the ASTM Fellow. Mr. Kaae is conducting research on the effect of hydrogen on the strain aging of steel for his doctoral dissertation.

T. A. Marshall, Jr., executive secretary, presented a résumé of the Annual Report of the Board of Directors.

Passing the Gavel:

At the conclusion of the business meeting, President Webber presented the gavel of authority to President-elect Williams who in turn bestowed the past-president's pin and certificate of service on Mr. Webber.

COMMITTEE REPORT SESSION

TUESDAY, JUNE 25, 11:30 A.M.

SESSION CHAIRMAN: W. R. WILLETS

Committee C-3 on Chemical-Resistant Non-Metallic Materials:

Report presented in the absence of the chairman by W. A. Whittier, ASTM staff, and the following actions taken:

Accepted as Tentative, Revisions in:

Test for Absorption and Apparent Porosity of Chemical-Resistant Mortars (C 413 - 58 T)
Test for Chemical Resistance of Mortars (C 267 - 62 T)

Adopted as Standard, Revisions in:

Rec. Practice for Use of Chemically Setting Silicate-Type Chemical-Resistant Mortars (C 397 - 62)
Rec. Practice for Use of Resin-Type Chemical-Resistant Mortars (C 399 - 60)
Spec. for Resin-Type Chemical-Resistant Mortars (C 395 - 60)

Committee C-8 on Refractories:

Report presented in the absence of the chairman by S. F. Etris, ASTM staff, and the following actions taken:

Accepted as Tentative, Revisions in:

Tests for Crushing Strength and Modulus of Rupture of Insulating Fire Brick at Room Temperature (C 93 - 54)
Classification of Silica Refractory Brick (C 416 - 60)

Adopted as Standard:

Classification of Steel Pouring Pit Refractories (C 435 - 61 T)
Classification of Refractory Granular Dolomite (C 468 - 61 T)

Adopted as Standard, Revision in:

Def. of Terms Relating to Refractories (C 71 - 62)

Committee C-13 on Concrete Pipe:

Report presented by P. W. Manson, chairman, and the following actions taken:

Accepted as Tentative:

Specs. for Nonreinforced Concrete Irrigation Pipe with Rubber-Type Gasket Joints (C 505 - 63 T)

Specs. for Reinforced Concrete Arch Culvert, Storm Drain, and Sewer Pipe (C 506 - 63 T)
Specs. for Reinforced Concrete Elliptical Culvert, Storm Drain, and Sewer Pipe (C 507 - 63 T)

Accepted as Tentative, Revisions in:

Spec. for Reinforced Concrete Culvert, Storm Drain, and Sewer Pipe (C 76 - 62 T)
Spec. for Precast Reinforced Concrete Manhole Risers and Tops (C 478 - 61 T)
Spec. for Joints for Circular Concrete Sewer and Culvert Pipe, Using Flexible, Watertight, Rubber-Type Gaskets (C 443 - 62 T)
Tests for Determining Physical Properties of Concrete Pipe or Tile (C 497 - 62 T)
Spec. for Cast-in-Place Nonreinforced Concrete Irrigation Closed Conduit (C 477 - 61 T)
Spec. for Concrete Sewer Pipe (C 14 - 59)

Adopted as Standard, Revisions in:

Spec. for Concrete Sewer Pipe (C 14 - 59)
Spec. for Concrete Pipe for Irrigation of Drainage (C 118 - 60)
Spec. for Concrete Drain Tile (C 412 - 60)

Committee C-16 on Thermal Insulating Materials:

Report presented in the absence of the chairman by E. C. Shuman, membership secretary, and the following actions taken:

Accepted as Tentative:

Test for Thermal Conductivity of Materials by Means of the Heat Flow Meter (C 518 - 63 T)
Test for Density of Fibrous Loose Fill Building Insulations (C 519 - 63 T)
Test for Density of Granular Loose Fill Insulations (C 520 - 63 T)
Spec. for Vermiculite Loose Fill Insulation (C 516 - 63 T)
Spec. for Diatomaceous Earth Block and Pipe Thermal Insulation (C 517 - 63 T)

Rec. Practice for Determining Nesting Pipe Sizes of Preformed Insulation for Pipes (IPS System) (C 521 - 63 T)

Accepted as Tentative, Revisions in:

Tests for Water Vapor Transmission of Materials Used in Building Construction (C 355 - 59 T)
Spec. for Mineral Fiber Molded-Type Pipe Insulation for Elevated Temperatures (C 281 - 58 T)
Spec. for Mineral Fiber Molded-Type Pipe Insulation for Low Temperatures (C 300 - 52 T)
Spec. for Mineral Fiber Block or Board Thermal Insulation for Low Temperatures (C 378 - 60 T)
Spec. for Mineral Fiber Batt Insulation (Industrial Type) (C 262 - 55 T)

Adopted as Standard, Revisions in:

Spec. for Mineral Fiber Block or Board Thermal Insulation for Elevated Temperatures (C 392 - 60)
Test for Thermal Conductivity of Materials by Means of the Guarded Hot Plate (C 177 - 45)

Tentatives Withdrawn:

Spec. for Diatomaceous Earth Thermal Insulation for Pipes (C 334 - 59 T)
Spec. for Diatomaceous Earth Block-Type Thermal Insulation (C 333 - 59 T)

Committee C-18 on Natural Building Stones:

Report presented in the absence of the chairman by W. F. Mullen, secretary, and accepted as a report of progress. The committee withdrew from the preprinted report the recommendations for withdrawal of Tentative Method of Test for Combined Effect of Temperature Cycles and Weak Salt Solutions on Natural Building Stones (C 218 - 48 T) and Tentative Specification for Structural Granite (C 422 - 58 T).

Committee C-20 on Acoustical Materials:

Report presented in the absence of the chairman by W. A. Jack, and the following actions taken:

Accepted as Tentative:

Test for Air Flow Resistance of Acoustical Materials (C 522 - 63 T)
Test for Light Reflectance of Acoustical Materials by the Integrating Sphere Reflectometer (C 523 - 63 T)

Committee C-22 on Porcelain Enamel:

Report presented in the absence of the chairman by Michael Bozsin and the following actions taken:

Adopted as Standard:

Def. of Terms Relating to Porcelain Enamel (C 286 -61 T)

Accepted as Tentative, Revisions in:

Def. of Terms Relating to Porcelain Enamel (C 286 - 61 T)

The revised definition for Pinhole, Pinholing, and the new definitions for Glass-Coated Steel' Glass-Lined Steel, Glassed Steel and One-Coat Ware, One-Coat Work, which were originally recommended for adoption as standard, were accepted for publication as tentative at the request of Committee C-22.

Committee C-25 on Ceramics for Electronics:

Report presented in the absence of the chairman by S. F. Etris, ASTM staff, and the following actions taken:

Accepted as Tentative:

Test for Ferrimagnetic Resonance Linewidth and Gyromagnetic Ratio of Nonmetallic Magnetic Materials (C 524 - 63 T)
Test for Complex Dielectric Constant of Nonmetallic Magnetic Materials at Microwave Frequencies (C 525 - 63 T)
Test for Nonmetallic Magnetic Cores to be Used in a Coincident Current Memory with a Two-to-One Selection Ratio Operating Under Full Switching Conditions (C 526 - 63 T)
Test for Saturation Magnetization or Saturation Induction of Nonmetallic Magnetic Materials (C 527 - 63 T)
Test for Compressive Strength of High-Strength Ceramic Materials (C 528 - 63 T)

SYMPOSIUM ON ADVANCES IN THE TECHNOLOGY OF STAINLESS STEELS AND RELATED ALLOYS[2a] (*Cont.*)

TUESDAY, JUNE 25, 2:00 P.M.

CO-CHAIRMEN: F. L. LAQUE
M. G. FONTANA

Experiences in Critical Applications

Criteria for the Selection of Stainless Steels for Service Between 1600 and 2000 F—K. G. Brickner, G. A. Ratz, and R. F. Domagala, presented by Mr. Brickner.

The Effects of Neutron Flux and Reactor Environments on Stainless Steels—S. H. Bush and J. C. Tobin, presented by Mr. Bush.

Evaluation and Applications of Stainless Steels in Cryogenic Environments—A. Hurlich and W. G. Scheck, presented by Mr. Hurlich.

Experiences with Stainless Steels in Utility Power Plants—G. E. Lien, presented by the author.

(*Continued at 7:30 p.m.*)

SYMPOSIUM ON THE CHEMICAL AND PHYSICAL EFFECTS OF HIGH-ENERGY RADIATION ON INORGANIC SUBSTANCES[6] (*Cont.*)

TUESDAY, JUNE 25, 2:00 P.M.

SESSION CHAIRMAN: E. S. FREEMAN

Radiation-Induced Decomposition of Inorganic Solids—E. R. Johnson, presented by the author.

Role of Electron Attachment in Radiolysis of Ionic Solids—J. Cunningham, presented by the author.

Dynamics of Low-Energy Radiation Damage Processes in a Body-Centered Cubic Lattice Simulating α-Iron—C. Erginsoy, presented by the author.

The Effect of Ionizing Radiation on the Optical and Electrical Properties of the Alkali Halides—B. S. H. Royce, presented by the author.

SESSION ON FATIGUE (*Cont.*)

TUESDAY, JUNE 25, 2:00 P.M.

SESSION CHAIRMAN: F. B. STULEN

Techniques for Axial Fatigue Testing of Sheet Materials Down to −423 F—R. D. Keys and F. R. Schwartzberg, presented by Mr. Keys.

An Evaluation of the Influence of Prestressing on Fatigue Limit—G. W. Brown and C. E. Work, presented by Mr. Work.

A Study of Fatigue Life Under Random Loading—H. A. Leybold and E. C. Naumann, presented by Mr. Leybold.

Effect of Shape of Cross-Section on Bending Fatigue Strength of Steel—M. R. Raghavan, presented by J. Marin.

COMMITTEE REPORT SESSION

TUESDAY, JUNE 25, 4:30 P.M.

SESSION CHAIRMAN: G. M. KLINE

Committee B-4 on Metallic Materials for Thermostats and for Electrical Resistance, Heating, and Contacts:

Report presented in the absence of the chairman by Leonard Mayeron, vice-chairman, and accepted as a report of progress.

Committee B-6 on Die-Cast Metals and Alloys:

Report presented by W. Babington, chairman, and the following actions taken:

Adopted as Standard:

Spec. for Zinc-Base Alloy Die Castings (B 86 – 57 T)
Spec. for Zinc-Base Alloys in Ingot Form for Die Castings (B 240 – 57 T)

Committee B-8 on Electrodeposited Metallic Coatings and Related Finishes:

Report presented by C. H. Sample, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Electrodeposited Coatings of Multi-Layer Nickel Plus Chromium on Zinc and Zinc-Base Alloys (B 405 – 63 T)

Accepted as Tentative, Revisions in:

Def. of Terms Relating to Electroplating (B 374 – 62 T)
Spec. for Electrodeposited Coatings of Multi-Layer Nickel Plus Chromium on Steel (B 375 – 61 T)
Test for Sealing of Anodically Coated Aluminum (B 136 – 45) (Standard reverted to tentative)

Committee C-4 on Clay Pipe:

Report presented in the absence of the chairman by R. H. Welles, vice-chairman, and accepted as a report of progress.

Committee C-7 on Lime:

Report presented in the absence of the chairman by K. A. Gutschick, secretary, and the following actions taken:

Adopted as Standard:

Spec. for Quicklime and Hydrated Lime for Water Treatment (C 53 – 52 T)
Spec. for Quicklime and Hydrated Lime for Sand-Lime Products (C 415 – 58 T)
Spec. for Quicklime and Hydrated Lime for Hypochlorite Bleach Manufacture (C 433 – 59 T)

Committee C-17 on Asbestos-Cement Products:

Report presented by W. V. Friedlaender, chairman, and the following actions taken:

Adopted as Standard:

Methods of Sampling and Testing Asbestos-Cement Flat Sehets, Roofing and Siding Shingles, and Clapboards (C 459 – 60 T)
Def. of Terms Relating to Asbestos-Cement and Related Products (C 460 – 60 T)

Committee D-8 on Bituminous and Organic Materials for Roofing, Waterproofing, and Related Building or Industrial Uses:

Report presented by W. C. Cullen, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Asphalt-Impregnated Glass Fiber Mat (Felt) (D 2178 – 63 T)

Adopted as Standard:

Test for Sieve Analysis of Granular Mineral Surfacing for Asphalt Roofing and Shingles (D 451 – 61 T)
Test for Sieve Analysis of Nongranular Mineral Surfacing for Asphalt Roofing and Shingles (D 452 – 61 T)
Test for Moisture in Mineral Aggregate for Use on Built-Up Roofs (D 1864 – 61 T)
Test for Hardness of Mineral Aggregate for Use on Built-Up Roofs (D 1865 – 61 T)
Spec. for Woven Glass Fabrics Treated with Bituminous Substances for Use in Waterproofing (D 1668 – 61 T)
Spec. for Mineral Aggregate for Use on Built-Up Roofs (D 1863 – 61 T)

Committee D-24 on Carbon Black:

Report presented by J. L. Hutson, chairman, and accepted as a report of progress.

Committee D-28 on Activated (Active) Carbon:

Report presented in the absence of the chairman by S. F. Etris, ASTM staff, and accepted as a report of progress.

Committee E-6 on Methods of Testing Building Constructions:

Report presented by R. F. Legget, chairman, and accepted as a report of progress.

Committee E-9 on Fatigue:

Report presented by H. J. Grover, chairman, and accepted as a report of progress.

Committee E-16 on Sampling and Analysis of Metal Bearing Ores and Related Materials:

Report presented by J. L. Hague, chairman, and accepted as a report of progress.

Committee F-1 on Materials for Electron Tubes and Semiconductor Devices:

No action was taken at the Annual Meeting on the Report of Committee F-1 in the absence of a quorum at the session when the report was scheduled for presentation. All recommendations in the report relate to tentatives and they will be submitted to the Administrative Committee on Standards for action on behalf of the Society.

GILLETT MEMORIAL LECTURE[7]

TUESDAY, JUNE 25, 5:00 P.M.

SESSION CHAIRMAN: PRESIDENT A. C. WEBBER

Twelfth Gillett Memorial Lecture:

President Webber opened the session by giving a brief statement of the purpose of the lecture, which is sponsored jointly by the ASTM and the Battelle Memorial Inst. to perpetuate the memory of Horace W. Gillett, one of America's leading technologists, the first director of the Institute, and for many years a very active worker in the Society, through the presentation of a lecture on a subject pertaining to the development, testing, evaluation, and application of metals.

President Webber called upon B. D. Thomas, President, Battelle Memorial Inst., who introduced J. B. Austin, administrative vice-president, research and technology, United States Steel Corp. Mr. Austin spoke on the subject "Trends in the Metallurgy of Low-Alloy, High-Yield-Strength Steels." He described improvements that have been made in structural steels since the low-alloy, high-strength steels were first introduced 30 years ago. Mr. Austin discussed the following new types of steels: low-carbon martensites, weldable alloy steels, and maraging and zerolled steels. In a look at the future, he explored the idea that future generations of metallurgists will make progress toward the ultimate goal, a steel with a strength approaching the binding force between iron atoms.

President Webber expressed appreciation to Mr. Austin for his outstanding lecture and presented to him on behalf of the Society the H. W. Gillett Memorial Lecture Certificate.

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[7] To be issued as a separate publication.

SYMPOSIUM ON ADVANCES IN THE TECHNOLOGY OF STAINLESS STEELS AND RELATED ALLOYS[2a] (*Cont.*)

TUESDAY, JUNE 25, 7:30 P.M.

CO-CHAIRMEN: L. L. WYMAN
A. G. COOK

Alloy Development: Part II

Maraging Stainless Steels—C. G. Bieber and J. J. Moran, Jr., presented by title only.

Modified Type 316 Stainless Steel with Low Tendency to Form Sigma—C. E. Spaeder and K. G. Brickner, presented by Mr. Spaeder.

Effect of Composition and Section Size on Mechanical Properties of Some Precipitation-Hardening Stainless Steels—W. C. Clarke and H. W. Garvin, presented by Mr. Garvin.

New High-Strength Alloy Grades by Structure Control—F. H. Beck and E. A. Schoefer, presented by Mr. Schoefer.

The Heat Treatment of Modified Type 304 Stainless Steel—R. B. G. Yeo and T. E. Scott, presented by Mr. Yeo.

SESSION ON CEMENT AND CONCRETE

(*Sponsored jointly by Committees C-1 on Cement and C-9 on Concrete and Concrete Aggregates*)

TUESDAY, JUNE 25, 8:00 P.M.

SESSION CHAIRMAN: MILOS POLIVKA

Significance of Tests for Heat of Hydration of Cement—W. J. McCoy, presented by the author.

Significance of Tests for Fineness—B. E. Kester, presented by the author.

Significance of False Set Tests—J. L. Gilliland, presented by the author.

Significant Time of Set Studies and Their Possible Relation to Current Time of Set Problems—E. E. Berger, presented by the author.

A Flow Test Involving Vibration for the Study of Plasticity and Workability of Pastes and Mortars—L. A. Weinland and S. L. Weinland., presented by Mr. L. A. Weinland.

The Development of Precision Statements for Several ASTM Test Methods—S. J. Hanna and J. F. McLaughlin, presented by Mr. Hanna.

Heat Curing of Sand, Lime, and Fly Ash Mixtures—M. Mateos, presented by title only

OPEN FORUM ON DISTRICT OBJECTIVES

TUESDAY, JUNE 25, 8:00 P.M.

SESSION CHAIRMAN: L. A. O'LEARY

Sponsored by the Administrative Committee on District Activities, this Forum was based on a questionaire circulated to all District Council Officers and Councilors. The purpose of the Forum, to which all members and committee members were invited, was to determine the purpose of ASTM Districts and what their objectives are, and how the Districts may be used more effectively.

SYMPOSIUM ON RECENT RESEARCH ON BITUMINOUS MATERIALS[8]

(*Sponsored by Committee D-8 on Bituminous and Other Organic Materials for Roofing, Waterproofing, and Related Building or Industrial Uses*)

WEDNESDAY, JUNE 26, 9:00 A.M.

SESSION CHAIRMAN: S. H. GREENFELD

The Effect of Carbon-Arc Intensity on Asphalt Oxidation—J. R. Wright, P. G. Campbell, and T. L. Friedinger, presented by Mr. Wright.

Alkylation of Asphaltenes—J. C. Illman and T. J. Deahl, presented by Mr. Illman.

Composition as Related to Manufacturing and Weathering of Roofing Asphalt—L. W. Corbett and R. E. Swarbrick, presented by Mr. Corbett.

The Prediction of Coating Grade Asphalt Weatherability from Asphaltene Dispersibility Data—T. K. Jones, presented by the author.

(*Continued at 2:30 p.m.*)

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[8] Issued as *ASTM STP 347*.

SYMPOSIUM ON SPACE RADIATION EFFECTS[9]

(Sponsored by Committee E-10 on Radioisotopes and Radiation Effects)

WEDNESDAY, JUNE 26, 9:00 A.M.

SESSION CHAIRMAN: J. W. KELLER

Nuclear Environment in Space and Its Simulation

The Radiation Environment in Space—J. W. Keller, presented by the author.

The Spectral Intensity Variations with Time in Specific Solar Flare Events and Radiation Doses—F. Foelsche, presented by the author.

Computer Codes for Space Radiation Effects—D. L. Dye, presented by the author.

Cosmic Ray Induced Stable and Radioactive Nuclides in Meteorites—M. E. Lipschutz, presented by the author.

Vacuum Systems Designed for Dynamic Studies of the Engineering Properties of Materials During Exposure to Nuclear Reactor Irradiation—O. H. Hill, E. E. Kerlin, and E. T. Smith, presented by Mr. Hill.

Experimental Techniques of Proton Accelerator Bombardment for Simulating Permanent Space Radiation Damage to Transistors and Diodes—R. R. Brown, presented by the author.

(Continued at 2:00 p.m. and on Thursday, June 27, at 9:00 a.m.)

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[9] Issued as *ASTM STP 363.*

SYMPOSIUM ON RECENT DEVELOPMENTS IN ADHESION SCIENCE[10]

(Sponsored by Division of Materials Sciences)

WEDNESDAY, JUNE 26, 9:00 A.M.

SESSION CHAIRMAN: F. H. WETZEL

The Application of Fracture Mechanics to Adhesive Joints—E. J. Ripling, S. Mostovoy, and R. L. Patrick, presented by Mr. Mostovoy.

Determination of the Nature of Intermolecular Forces Between Adjacent Materials by Surface Chemical Techniques—F. M. Fowkes, presented by the author.

Analysis of Fiber-Elastomer Adhesion Variables—H. T. Patterson, presented by the author.

(Continued at 2:30 p.m.)

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[10] Issued with the Symposium on Recent Advances in Adhesives Technology as *ASTM STP 360.*

TECHNICAL SESSION ON SKID RESISTANCE[11] *(Informal)*

(Sponsored by Committee E-17 on Skid Resistance)

WEDNESDAY, JUNE 26, 9:00 A.M.

SESSION CHAIRMAN: J. E. GRAY

Movie of Tappahannock Correlation Study, prepared by the State of Tennessee.

Comparison of Several Methods of Measuring Road Surface Slipperiness—Tappahannock, Va., 1962—J. H. Dillard and D. C. Mahone.

Prepared discussions by B. Wehner, R. S. Rizenbergs, J. E. Bell, H. W. Kummer, W. Gartner, Jr., and T. H. Forrer.

Open discussion.

(Continued at 2:00 p.m.)

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[11] Issued as *ASTM STP 366.*

TECHNICAL SESSION ON ELECTRON METALLOGRAPHY (*Informal*)

(*Sponsored by Subcommittee XI on Electron Metallography of Metals of Committee E-4 on Metallography*)

WEDNESDAY, JUNE 26, 9:30 A.M.

SESSION CHAIRMAN: W. R. LASKO

Investigation of Room-Temperature Fracture in SM Nickel-Base Alloys—R. W. Hertzberg and J. A. Ford.

Application of Conducting Mask for Thinning Metallic Foils for Electron Transmission Microscopy—T. A. Despres.

The Use of Combined Instrumental Techniques in Metallurgical Problems—J. R. Mihalisin and C. Cuff.

Electron Diffraction Analysis of Molybdenum Surfaces Produced by Fracture—C. S. Tufts.

The Nature of MC_6 Carbide in High Speed Steel—P. K. Koh and H. Nikkel.

Progress Report of Task Group D, Electron Probe Microanalysis—L. S. Birks.

Specimen Damage During Spark Plane Cutting Steel—A. Szirmae and R. M. Fisher.

AWARDS LUNCHEON

WEDNESDAY, JUNE 26, 12 NOON

TOASTMASTER: PAST PRESIDENT F. L. LAQUE

INVOCATION: HARVEY BENNET, PASTOR, FIRST PRESBYTERIAN CHURCH, ATLANTIC CITY, N. J.

There were 274 members and guests at the Awards Luncheon.

Conferral of Committee Awards:

The C. A. Hogentogler Award of Committee D-18 on Soils for Engineering Purposes was made to R. K. Bernhard for his paper "Biaxial Stress Fields in Noncohesive Soils Subjected to Vibratory Loads." He was introduced to the president for the presentation by W. G. Holtz, chairman of Committee D-18.

The Max Hecht Award of Committee D-19 on Industrial Water was presented to J. F. James Thomas, a member of the committee, for his outstanding work in the field of industrial water. The recipient was introduced to the president by Frank E. Clarke, vice-chairman of Committee D-19.

The Sanford E. Thompson Award of Committee C-9 on Concrete and Concrete Aggregates was given to D. L. Bloem, R. D. Gaynor, and J. R. Wilson for their paper, "Testing Uniformity of Large Batches of Concrete." The recipients were introduced to the president by Bryant Mather, chairman of Committee C-9.

Conferral of Society Awards:

The Walter C. Voss Award—to an engineer or scientist, inside or outside the Society, who has contributed notably to the knowledge in the field of building technology, with emphasis upon materials used which constitute significant advances or innovations—was presented to Douglas E. Parsons. The recipient was introduced to the president for the conferral of the award by Werner H. Gumpertz, chairman of the award committee.

The Frank E. Richart Award—to a member of the Society who has made notable contributions to the work of the Society in the field of concrete and concrete aggregates—was presented to

Kenneth B. Woods. The recipient was introduced to the president by Stanton Walker, a member of the award committee.

The Richard L. Templin Award—for a meritorious paper describing new testing methods and apparatus—was given to F. Schwartzberg and R. Markovich (deceased) for their paper, "Testing Techniques and Evaluation of Materials for Use at Liquid Hydrogen Temperature." Mr. Schwartzberg was introduced to the president by R. H. Heyer, chairman of the award committee.

The Charles B. Dudley Medal—to the author or authors of a paper published by the Society, of outstanding merit, constituting an original contribution on research in engineering materials—was presented to M. R. Meyerson, T. R. Young, and W. R. Ney for their paper, "The Development of More Stable Gage Blocks." The recipients were introduced to the president by C. H. Scholer, chairman of the medal committee.

Awards of Merit.—The chairman of the Award of Merit Committee, F. C. Burk, read brief citations and introduced the recipients present to President Webber who conferred on them the certificate of Award of Merit. Those receiving an Award of Merit were:

William M. Bertholf
Francisco Chairez
Ronald E. Copeland
Donald I. Finch
Delvan Halloran
Harry K. Hammond
Waldemar C. Hansen
Simon H. Ingber
Daniel E. Krause
M. A. Pinney
Charles E. Proudley
Lloyd F. Rader
Earle E. Schumacher
Raymond B. Smith
J. Vernon Steinle
Joseph A. Truitt
Clarence Zischkau

Messrs. Pinney, Schumacher, and Steinle were not present. The certificates of Messrs. Pinney and Schumacher were received on their behalf by other members of the Society.

The ASTM Award to Executives—to an executive who through his outstanding interest and support has furthered the accomplishments of ASTM—was presented to Alexander C. Monteith, Vice President, Electric Utility and Marine Group of the Westinghouse Electric Corp., Pittsburgh. Mr. Monteith delivered a brief address in response.

SYMPOSIUM ON RECENT DEVELOPMENTS IN ADHESION SCIENCE[10]

(*Cont.*)

WEDNESDAY, JUNE 26, 2:30 P.M.

SESSION CHAIRMAN: F. W. REINHART

Work of Adhesion Cleavage Stress at Interfaces Between Solids and a Pressure Sensitive Adhesive—C. A. Dahlquist, presented by the author.

Studies on the Chemical Nature of Polymer to Metals Adhesion—A. F. Lewis and L. J. Forrestal, presented by Mr. Lewis.

SYMPOSIUM ON SPACE RADIATION EFFECTS[9] (*Cont.*)

WEDNESDAY, JUNE 26, 2:00 P.M.

SESSION CHAIRMAN: P. H. MILLER

Space Radiation Effects on Components and Systems

Displacement Radiation Effects on Components and Systems—P. H. Miller, Jr., and V. A. J. van Lint, presented by Mr. Miller.

Correlation of Proton to Neutron and Electron to Photon Radiation Damage in Transistors and Diodes—G. L. Keister, presented by the author.

Annealing of Radiation Damage in Space—D. Binder, presented by the author.

Effects of Space Nuclear Radiation upon Transistors—L. B. Gardner and J. R. Coss, presented by P. H. Miller.

The Effects of Neutron Irradiation on the Magnetic Properties of Ferromagnetic Bulk Alloys and Thin Films—A. I. Schindler and C. M. Williams, presented by Mr. Schindler.

(*Continued on Thursday, June 27, at 9:00 a.m.*)

TECHNICAL SESSION ON SKID RESISTANCE[11] (*Cont.*)

WEDNESDAY, JUNE 26, 2:00 P.M.

SESSION CHAIRMAN: R. G. WILCOX

Movie of Tire Hydroplaning—Hazards to Aircraft Operation as prepared by NASA.

Hazards of Tire Hydroplaning—W. B. Horne and J. W. Leland.

Improved Instrumentation for Determining Pavement Coefficient by Means of Vehicle Locked Wheel Skids—F. D. Bardwell.

Panel discussion—Standardization of Trailers:

R. H. Sawyer, Moderator—Summary of present status of trailers pointing out specific advantages and limitations of types of trailers and presentation of ideas on a prototype trailer.

H. W. Kummer—Discussion of requirements of a standard trailer or trailers for research and general field testing, including how existing trailers might satisfy these needs and what modifications might be needed on types and, if necessary, individual trailers.

A. Y. Casanova—Discussion of a uniform operating procedure for existing trailers. This will concern preparation for testing, calibration checks, selection of test speeds, wheel track (left or right) in which the test should be conducted, length of wheel lock, trace evaluation, etc.

Open discussion.

SYMPOSIUM ON RECENT RESEARCH ON BITUMINOUS MATERIALS[8] (*Cont.*)

WEDNESDAY, JUNE 26, 2:30 P.M.

SESSION CHAIRMAN: W. C. CULLEN

Behavior of Asphalts During Prolonged Weathering, as Influenced by the Relative Homogeneity of Their Internal Structure—G. L. Oliensis, presented by the author.

Some Engineering Properties of Built-Up Roofing—P. M. Jones, presented by the author.

New Concepts in the Blistering of Asphalt Shingles—R. S. Knight, R. B. Platt, and D. J. Smith, presented by Mr. Smith.

The Effect of Heat on Oxidized Asphalts—J. A. Wronka, presented by the author.

COMMITTEE REPORT SESSION

WEDNESDAY, JUNE 26, 4:30 P.M.

SESSION CHAIRMAN: W. L. GLOWACKI

Committee C-9 on Concrete and Concrete Aggregates:

Report presented in the absence of the chairman by Paul Klieger, secretary, and the following actions taken:

Accepted as Tentative:

Test for Creep of Concrete in Compression (C 512 - 63 T)

Method for Securing, Preparing, and Testing Specimens from Hardened Lightweight Insulating Concrete for Compressive Strength (C 513 - 63 T)

Accepted as Tentative, Revisions in:

Test for Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar - Bar Method) (C 227 - 61 T)

Test for Potential Reactivity of Aggregates (Chemical Method) (C 289 - 61 T)

Test for Effectiveness of Mineral Admixtures in Preventing Excessive Expansion of Concrete Due to the Alkali-Aggregate Reaction (C 441 - 62 T)

Test for Measuring Mortar-Making Properties of Fine Aggregate (C 87 - 62 T), with the addition of the following new Section 3(*b*):

(*b*) Tamper, made of a nonabsorptive, nonabrasive, nonbrittle material such as a rubber compound having a Shore A durometer hardness of 80 ± 10 or seasoned oak wood rendered nonabsorptive by immersion for 15 min in paraffin at approximately 200 C (392 F) and having a cross-section of ½ by 1 in. and a convenient length (5 or 6 in.). The tamping face shall be flat and at right angles to the length of the tamper.

Test for Lightweight Pieces in Aggregate (C 123 - 57 T)

Spec. for Raw or Calcined Natural Pozzolans for Use as Admixtures in Portland Cement Concrete (C 402 - 62 T)

Test for Resistance of Concrete Specimens to Rapid Freezing and Thawing in Water (C 290 - 61 T)

Test for Resistance of Concrete Specimens to Slow Freezing and Thawing in Water or Brine (C 292 - 61 T)

Method of Testing Air-Entraining Admixtures for Concrete (C 233 - 60 T), with a further change in Section 2(*c*) by deleting in the first sentence the words "from which specimens will be made for test for resistance to freezing and thawing as specified in Section 6(*b*)."

Spec. for Air-Entraining Admixtures for Concrete (C 260 - 60 T)

Spec. for Fly Ash for Use as an Admixture in Portland Cement Concrete (C 350 - 60 T)

Spec. for Chemical Admixtures of Concrete (C 494 - 62 T)

Test for Time of Setting of Concrete Mixtures by Penetration Resistance (C 403 - 62 T)

Test for Compressive Strength of Molded Concrete Cylinders (C 39 - 61)

At the session Committee C-9 requested that the revision of Method C 39 be changed from immediate adoption to a tentative revision.

Adopted as Standard:

Test for Static Young's Modulus of Elasticity and Poisson's Ratio in Compression of Cylindrical Concrete Specimens (C 469 - 61 T)

Test for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 88 - 61 T)

Test for Sieve or Screen Analysis of Fine and Coarse Aggregates (C 136 - 61 T)

Test for Ball Penetration in Fresh Portland Cement Concrete (C 360 - 55 T)

Spec. for Concrete Aggregates (C 33 - 61 T)

Spec. for Waterproof Paper for Curing Concrete (C 171 - 60 T)

Test for Materials Finer Than No. 200 Sieve in Mineral Aggregates by Washing (C 117 - 62 T)

The recommendation that Method C 117 - 62 T be adopted as standard did not receive the concurrence of Committee D-4 on Road and Paving Materials, joint sponsor for the method

Adopted as Standard, Revisions in:

Test for Weight Per Cubic Foot, Yield, and Air Content (Gravimetric) of Concrete (C 138 - 44)

Test for Abrasion of Graded Coarse Aggregate by Use of the Deval Machine (D 289 - 55) (Jointly with Committee D-4)

Spec. for Ready-Mixed Concrete (C 94 - 62):

(*a*) Immediate adoption of revisions of Sections 3(*d*) and 7(*b*).

(*b*) Adoption of tentative revision of Section 9(*c*), issued June, 1961, including editorial revision of Table I.

Committee C-12 on Mortars for Unit Masonry:

Report presented by H. C. Plummer, chairman, and the following action taken:

Adopted as Standard:

Spec. for Mortar and Grout for Reinforced Masonry (C 476 - 61 T)

Committee D-1 on Paint, Varnish, Lacquer, and Related Products:

Report presented by W. T. Pearce, chairman, and the following actions taken:

Accepted as Tentative:

Test for Acetaldehyde Content of Vinyl Acetate (D 2191 - 63 T)

Test for Purity of Acetaldehyde (D 2192 - 63 T)

Test for Hydroquinone in Vinyl Acetate (D 2193 - 63 T)

Test for Concentration of Formaldehyde Solutions (D 2194 - 63 T)

Test for Pentaerythritol (D 2195 - 63 T)

Method for Determination of Low Quantities of Mercury in Paint (D 2206 - 63 T)

Test for Perspiration Resistance of Organic Coatings (D 2204 - 63 T)

Objection to this perspiration test was presented at the session by Sam Tour. While the session accepted the test for publication as tentative, Mr. Tour's objections will be published in the *Proceedings* with the discussion in support of the method that had been prepared by Committee D-1.

Test for Adhesion of Coatings of Paint, Varnish, Lacquer, and Related Products with Scrape-Adhesion Apparatus (D 2197 - 63 T)

Test for Rheological Properties of Non-Newtonian Materials (D 2196 - 63 T)

Test for Slump of Oil and Resin-Base Caulking Compounds (D 2202 - 63 T)

Test for Staining of Oil and Resin-Base Caulking Compounds (D 2203 - 63 T) with the addition of the following alternate procedure:

Alternate Procedure.—If the sample of caulking to be tested is accompanied by a block of masonry (brick, marble, limestone, etc.) that is to be used on the job in question, in lieu of the standard stain test, the following alternate test may be used in its stead:

Dry duplicate specimens of the masonry sample (3½ by 3½ in. by ¾ in.) in an oven at 220 ± 3 F for 24 hr and cool to room temperature. Place a round pat of the caulking compound 1½ in. in diameter and about ¼ in. high on the surface of each block. Expose the blocks in air for 5 days at 77 ± 2 F. At the end of this period, measure and record the width of the stain (if any) to the nearest 0.1 mm. Take four readings at 90 deg points around each pat. Record the average of eight readings.

Remove the caulking with a putty knife and break blocks in two, approximately through the middle of the stained area, by a sharp blow with a small hammer. Measure and record the depth of penetration of the stain into the block, taking three readings on each broken piece—two on the ends and one in the middle of the stained surface. Average the twelve readings and record the results.

The average of the stain values obtained on the surface of the block and the average value obtained into the depth of the block is the final stain value.

Test for Stain Removal from Multicolor Lacquers (D 2198 - 63 T)

Method for Measurement of Plasticizer Migration from Vinyl Fabrics to Lacquers (D 2199 - 63 T)

Method for Preparation of Hot-Dipped Non-passivated Galvanized Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products (D 2201 - 63 T)

Specs. for Vinyl Acetate (D 2190 - 63 T)

Specs. for Acetaldehyde (D 2189 - 63 T)

Rec. Practices for Testing Traffic Paints (D 2205 - 63 T)

Pictorial Surface Preparation Standards for Painting Steel Surfaces (D 2200 - 63 T)

Accepted as Tentative, Revisions in:

Test for Color of Transparent Liquids (Gardner Color Scale) (D 1544 - 58 T)

Spec. for Raw Castor Oil (D 960 - 52) (Standard reverted to tentative)

Adopted as Standard:

Spec. for Distilled Coconut Fatty Acids (D 1841 - 61 T)

Spec. for Distilled Corn Fatty Acids (D 1842 - 61 T)

Spec. for Fractionated and Distilled Cottonseed Fatty Acids (D 1843 - 61 T)

Spec. for Asbestos-Cement Shingle Blanks to be Used as Panels in Weathering Tests of Latex and Emulsion Paints (D 1911 - 61 T)

Test for Hydroxyl Value of Fatty Oils and Acids (D 1957 - 61 T)

Test for Color Change of White Architectural Enamels (D 1543 - 60 T)

Test for Package Stability of Latex Paint (D 1849 - 61 T)

Test for Flash Point of Volatile Flammable

Materials by Tag Open-Cup Apparatus (D 1310 - 62 T)
Method for Qualitative Determination of Fish Oil in Drying Oils and Drying Oil Fatty Acids (D 1724 - 60 T)
Method for Preparation of Aluminum Alloy Panels for Testing Paint, Varnish, Lacquer, and Related Products (D 1733 - 60 T)
Method of Making and Preparing Concrete and Masonry Panels for Testing Paint Finishes (D 1734 - 60 T)
Methods for Chemical Analysis of Dry Mercuric Oxide Pigment (D 284 - 57 T)
Methods for Chemical Analysis of Zinc Dust (Metallic Zinc Powder) (D 521 - 50 T)
Methods for Chemical Analysis of White Titanium Pigments (D 1394 - 56 T)
Methods for Chemical Analysis of Basic Lead Silico-Chromate (D 1844 - 61 T)
Method of Reporting Paint Film Failures Characteristic of Exterior Latex Paints (D 1848 - 61 T)

Adopted as Standard, Revisions in:

Methods of Chemical Analysis of Dry Red Lead (D 49 - 44)
Methods of Analysis of Barium Sulfate Pigments (D 715 - 45)
Methods of Analysis of Mica Pigments (D 716 - 45)
Methods of Analysis of Magnesium Silicate Pigment (D 717 - 45)
Methods of Analysis of Aluminum Silicate Pigment (D 718 - 45)
Methods of Analysis of Diatomaceous Silica Pigment (D 719 - 51)
Test for Viscosity of Transparent Liquids by Bubble Time Method (D 1545 - 60)
Test for Distillation Range of Lacquer Solvents and Diluents (D 1078 - 58)

The committee withdrew from the preprinted report the proposed Tentative Method of Test for Fatty Acid Composition by Gas Chromatography and the proposed Tentative Specifications for Tall Oil Fatty Acids.

Committee D-5 on Coal and Coke:

Report presented in the absence of the chairman by R. F. Abernethy, secretary, and the following actions taken:

Accepted as Tentative, Revisions in:

Test for Free-Swelling Index of Coal (D 720 - 57)
Method of Tumbler Test for Coke (D 294 - 50) (Standard reverted to tentative)

Committee D-18 on Soils for Engineering Purposes:

Report presented in the absence of the chairman by W. S. Housel and the following actions taken:

Accepted as Tentative:

Method for Calibration of Mechanical Laboratory Soil Compactors (D 2168 - 63 T)
Test for Unconfined Compressive Strength of Cohesive Soil (D 2166 - 63 T)
Test for Density of Soil in Place by the Rubber-Balloon Method (D 2167 - 63 T)

Accepted as Tentative, Revisions in:

Method for Soil Investigation and Sampling by Auger Borings (D 1452 - 56 T)
Penetration Test and Split-Barrel Sampling of Soils (D 1586 - 58 T)
Method for Thin-Walled Tube Sampling of Soils (D 1587 - 58 T)

Adopted as Standard:

Test for Moisture-Penetration Resistance Relations of Fine-Grained Soils (D 1558 - 58 T)
Test for Making and Curing Soil-Cement Compression and Flexure Test Specimens in the Laboratory (D 1632 - 59 T)
Test for Compressive Strength of Molded Soil-Cement Cylinders (D 1633 - 59 T)
Test for Compressive Strength of Soil-Cement Using Portions of Beams Broken in Flexure (Modified Cube Method) (D 1634 - 59 T)
Test for Flexure Strength of Soil-Cement Using Simple Beam with Third-Point Loading (D 1635 - 59 T)

Committee D-27 on Electrical Insulating Liquids and Gases:

Report presented by E. R. Thomas, chairman, and the following actions taken:

Adopted as Standard:

Test for Scavenger Content of Askarels (D 1701 - 59 T)
Test for Volume of Oil in Oil-Contaminated Askarels (D 1808 - 60 T)
Test for Specific Gravity of Askarels (D 1810 - 60 T)
Test for Inorganic Chlorides in Askarels (D 1821 - 60 T)
Test for Water in Insulating Oils by Extraction (D 1315 - 59 T)
Rec. Practice for Estimating Relative Temperature Limits of Dielectric Liquids (D 1805 - 60 T)

Continuity Specification for Electrical Insulating Oil for Low-Pressure Cable Systems (D 1818 - 60 T)

Continuity Specification for Electrical Insulating Oil for High-Pressure Cable Systems (D 1819 - 60 T)

Adopted as Standard, Revisions in:

Methods of Testing Askarels (D 901 - 62)

Committee E-1 on Methods of Testing:

Report presented in the absence of the chairman by P. J. Smith, secretary, and the following actions taken:

Accepted as Tentative:

Specs. and Methods of Test for Cover Glasses and Glass Slides for Use in Microscopy (E 211 - 63 T)

Accepted as Tentative, Revisions in:

Tests for Water Vapor Transmission of Materials in Sheet Form (E 96 - 53 T)

Rec. Practice for Operating Enclosed Carbon-Arc Type Apparatus for Artificial Light Exposure Tests (E 188 - 61 T)

Adopted as Standard:

Standard Microscope Objective Thread (E 210 - 63)

Def. of Terms Relating to Conditioning (E 41 - 60 T)

Spec. for Standard Atmospheres for Conditioning and Testing Materials (E 171 - 60 T)

Adopted as Standard, Revisions in:

Def. of Terms Relating to Methods of Mechanical Testing (E 6 - 62)

(*a*) Adopt as standard with revision tentative definitions for Set, Breaking Load, Fatigue, Stress Ratio, and Creep.

(*b*) Adopt as standard without revision tentative definition for Stress Cycles Endured.

(*c*) Immediate adoption as standard of definitions for Modulus of Rupture in Bending, and Modulus of Rupture in Torsion.

Committee F-3 on Gaskets:

Report presented by T. H. Redman, chairman, and accepted as a report of progress.

COMMITTEE REPORT SESSION

WEDNESDAY, JUNE 26, 4:30 P.M.

SESSION CHAIRMAN: T. J. DOLAN

Committee A-1 on Steel:

Report presented by J. J. Kanter, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Carbon Steel Eyebolts (A 489 - 63 T)

Spec. for Steel Castings for Highway Bridges (A 486 - 63 T)

Spec. for Low-Alloy Steel Castings Suitable for Pressure Service (A 487 - 63 T)

Spec. for Quenched-and-Tempered Steel Bolts and Studs (A 449 - 63 T)

Rec. Practice for Qualification of Procedures and Personnel for the Welding of Steel Castings (A 488 - 63 T)

Accepted as Tentative, Revisions in:

Spec. for General Requirements for Delivery of Rolled Steel Plates of Flange and Firebox Qualities (A 20 - 62 T)

Spec. for Welded and Seamless Steel Pipe (A 53 - 62 T)

Spec. for Seamless Carbon-Steel Pipe for High-Temperature Service (A 106 - 62 T)

Spec. for Black and Hot-Dipped (Galvanized) Welded and Seamless Steel Pipe for Ordinary Uses (A 120 - 62 T)

Spec. for Electric-Resistance-Welded Steel Pipe (A 135 - 62 T)

Spec. for Electric-Fusion-Welded Steel Pipe for High-Temperature Service (A 155 - 62 T)

Spec. for Carbon-Steel Castings Suitable for Fusion Welding for High-Temperature Service (A 216 - 60 T)

Spec. for Carbon-Steel Forgings for General Industrial Use (A 235 - 62 T)

Spec. for Alloy-Steel Forgings for General Industrial Use (A 237 - 62 T)

Spec. for Carbon-Steel Seamless Drum Forgings (A 266 - 62 T)

Spec. for Carbon-Steel Blooms, Billets, and Slabs for Forgings (A 273 - 58 T)

Spec. for Alloy-Steel Blooms, Billets, and Slabs for Forgings (A 274 - 58 T)

Spec. for Low-Carbon Steel Externally and Internally Threaded Standard Fasteners (A 307 - 61 T)

Spec. for Seamless and Welded Austenitic

Stainless Steel Pipe (A 312 - 62 T) (Jointly with Committee A-10)

Spec. for Alloy-Steel Bolting Materials for Low-Temperature Service (A 320 - 62 T)

Spec. for High-Strength Bolts for Structural Steel Joints, Including Suitable Nuts and Plain Hardened Washers (A 325 - 61 T)

Spec. for Seamless and Welded Steel Pipe for Low-Temperature Service (A 333 - 62 T), with the withdrawal from the preprinted report of changes in Section 12 and Tables II and III.

Spec. for Seamless and Welded Carbon and Alloy-Steel Tubes for Low-Temperature Service (A 334 - 62 T), with the withdrawal from the preprinted report of changes in Tables I, II, and V.

Spec. for Seamless Ferritic Alloy Steel Pipe for High-Temperature Service (A 335 - 62 T)

Spec. for Ferritic and Austenitic Steel Castings for High-Temperature Service (A 351 - 61 T)

Spec. for Quenched and Tempered Alloy Steel Bolts and Studs with Suitable Nuts (A 354 - 62 T)

Spec. for Electric-Fusion-Welded Austenitic Chromium-Nickel Alloy Steel Pipe for High-Temperature Service (A 358 - 62 T)

Spec. for Carbon and Alloy Steel Forgings for Pressure Vessel Shells (A 372 - 62 T)

Spec. for Seamless Austenitic Steel Pipe for High-Temperature Central-Station Service (A 376 - 62 T)

Spec. for Galvanized Steel Transmission Tower Bolts and Nuts (A 394 - 61 T)

Spec. for Seamless Ferritic Alloy-Steel Pipe Specially Heat-Treated for High-Temperature Service (A 405 - 62 T)

Spec. for Upholstery Spring Wire for Coiled Type Springs (A 407 - 57 T)

Spec. for Welded Large Outside Diameter Light-Wall Austenitic Chromium-Nickel Alloy Steel Pipe for Corrosive or High-Temperature Service (A 409 - 60 T)

Spec. for Upholstery Spring Wire for Zig-Zag Type and Square Formed Type and No-Sag Type Spring Units (A 417 - 57 T)

Spec. for Factory-Made Wrought Carbon Steel and Alloy Steel Welded Fittings of Seamless or Welded Construction for Low-Temperature Service (A 420 - 61 T)

Spec. for Centrifugally Cast Ferritic Alloy Steel Pipe for High-Temperature Service (A 426 - 62 T)

Spec. for General Requirements for Carbon, Ferritic Alloy, and Austenitic Alloy Steel Tubes (A 450 - 62 T) (Jointly with Committee A-10)

Spec. for Centrifugally Cast Austenitic Steel Pipe for High-Temperature Service (A 451 - 62 T) (Jointly with Committee A-10)

Spec. for Centrifugally Cast Austenitic Cold-Wrought Pipe for High-Temperature Service (A 452 - 62 T) (Jointly with Committee A-10)

Method for Ultrasonic Testing and Inspection of Turbine and Generator Steel Rotor Forgings (A 418 - 57 T)

Spec. for Hard-Drawn Steel Spring Wire (A 227 - 47) (Standard reverted to tentative)

Spec. for Steel Music Spring Wire (A 228 - 51) (Standard reverted to tentative)

Spec. for Oil-Tempered Steel Spring Wire (A 229 - 56) (Standard reverted to tentative)

Spec. for Carbon-Steel Valve Spring Quality Wire (A 230 - 47) (Standard reverted to tentative)

Spec. for Chromium-Vanadium Steel Spring Wire (A 231 - 41) (Standard reverted to tentative)

Spec. for Chromium-Vanadium Steel Valve Spring Quality Wire (A 232 - 47) (Standard reverted to tentative)

Spec. for Chromium-Silicon Steel Spring Wire (A 401 - 58) (Standard reverted to tentative)

The revision of the following tentative specifications, consisting of the addition of a new Section 3 on Basis of Purchase, was inadvertently listed in the preprinted report under Editorial Changes:

Spec. for Seamless Carbon Steel Boiler Tubes (A 83 - 62 T)

Spec. for Seamless Low-Carbon and Carbon-Molybdenum Steel Still Tubes for Refinery Service (A 161 - 62 T)

Spec. for Electric-Resistance-Welded Carbon Steel Boiler Tubes (A 178 - 62 T)

Spec. for Seamless Cold-Drawn Low-Carbon Steel Heat-Exchanger and Condenser Tubes (A 179 - 62 T)

Spec. for Seamless Carbon Steel Boiler Tubes for High-Pressure Service (A 192 - 62 T)

Spec. for Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes (A 199 - 62 T)

Spec. for Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service (A 200 - 62 T)

Spec. for Seamless Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 209 - 62 T)

Spec. for Seamless Medium-Carbon Steel Boiler and Superheater Tubes (A 210 - 62 T)

Spec. for Seamless Ferritic and Austenitic Alloy-Steel Boiler, Superheater, and Heat Exchanger Tubes (A 213 - 62 T)

Spec. for Electric-Resistance-Welded Carbon Steel Heat Exchanger and Condenser Tubes (A 214 - 62 T)

Continuity Specification for Electrical Insulating Oil for Low-Pressure Cable Systems (D 1818 – 60 T)

Continuity Specification for Electrical Insulating Oil for High-Pressure Cable Systems (D 1819 – 60 T)

Adopted as Standard, Revisions in:

Methods of Testing Askarels (D 901 – 62)

Committee E-1 on Methods of Testing:

Report presented in the absence of the chairman by P. J. Smith, secretary, and the following actions taken:

Accepted as Tentative:

Specs. and Methods of Test for Cover Glasses and Glass Slides for Use in Microscopy (E 211 – 63 T)

Accepted as Tentative, Revisions in:

Tests for Water Vapor Transmission of Materials in Sheet Form (E 96 – 53 T)

Rec. Practice for Operating Enclosed Carbon-Arc Type Apparatus for Artificial Light Exposure Tests (E 188 – 61 T)

Adopted as Standard:

Standard Microscope Objective Thread (E 210 – 63)

Def. of Terms Relating to Conditioning (E 41 – 60 T)

Spec. for Standard Atmospheres for Conditioning and Testing Materials (E 171 – 60 T)

Adopted as Standard, Revisions in:

Def. of Terms Relating to Methods of Mechanical Testing (E 6 – 62)

(*a*) Adopt as standard with revision tentative definitions for Set, Breaking Load, Fatigue, Stress Ratio, and Creep.

(*b*) Adopt as standard without revision tentative definition for Stress Cycles Endured.

(*c*) Immediate adoption as standard of definitions for Modulus of Rupture in Bending, and Modulus of Rupture in Torsion.

Committee F-3 on Gaskets:

Report presented by T. H. Redman, chairman, and accepted as a report of progress.

COMMITTEE REPORT SESSION

WEDNESDAY, JUNE 26, 4:30 P.M.

SESSION CHAIRMAN: T. J. DOLAN

Committee A-1 on Steel:

Report presented by J. J. Kanter, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Carbon Steel Eyebolts (A 489 – 63 T)

Spec. for Steel Castings for Highway Bridges (A 486 – 63 T)

Spec. for Low-Alloy Steel Castings Suitable for Pressure Service (A 487 – 63 T)

Spec. for Quenched-and-Tempered Steel Bolts and Studs (A 449 – 63 T)

Rec. Practice for Qualification of Procedures and Personnel for the Welding of Steel Castings (A 488 – 63 T)

Accepted as Tentative, Revisions in:

Spec. for General Requirements for Delivery of Rolled Steel Plates of Flange and Firebox Qualities (A 20 – 62 T)

Spec. for Welded and Seamless Steel Pipe (A 53 – 62 T)

Spec. for Seamless Carbon-Steel Pipe for High-Temperature Service (A 106 – 62 T)

Spec. for Black and Hot-Dipped (Galvanized) Welded and Seamless Steel Pipe for Ordinary Uses (A 120 – 62 T)

Spec. for Electric-Resistance-Welded Steel Pipe (A 135 – 62 T)

Spec. for Electric-Fusion-Welded Steel Pipe for High-Temperature Service (A 155 – 62 T)

Spec. for Carbon-Steel Castings Suitable for Fusion Welding for High-Temperature Service (A 216 – 60 T)

Spec. for Carbon-Steel Forgings for General Industrial Use (A 235 – 62 T)

Spec. for Alloy-Steel Forgings for General Industrial Use (A 237 – 62 T)

Spec. for Carbon-Steel Seamless Drum Forgings (A 266 – 62 T)

Spec. for Carbon-Steel Blooms, Billets, and Slabs for Forgings (A 273 – 58 T)

Spec. for Alloy-Steel Blooms, Billets, and Slabs for Forgings (A 274 – 58 T)

Spec. for Low-Carbon Steel Externally and Internally Threaded Standard Fasteners (A 307 – 61 T)

Spec. for Seamless and Welded Austenitic

Stainless Steel Pipe (A 312 - 62 T) (Jointly with Committee A-10)

Spec. for Alloy-Steel Bolting Materials for Low-Temperature Service (A 320 - 62 T)

Spec. for High-Strength Bolts for Structural Steel Joints, Including Suitable Nuts and Plain Hardened Washers (A 325 - 61 T)

Spec. for Seamless and Welded Steel Pipe for Low-Temperature Service (A 333 - 62 T), with the withdrawal from the preprinted report of changes in Section 12 and Tables II and III.

Spec. for Seamless and Welded Carbon and Alloy-Steel Tubes for Low-Temperature Service (A 334 - 62 T), with the withdrawal from the preprinted report of changes in Tables I, II, and V.

Spec. for Seamless Ferritic Alloy Steel Pipe for High-Temperature Service (A 335 - 62 T)

Spec. for Ferritic and Austenitic Steel Castings for High-Temperature Service (A 351 - 61 T)

Spec. for Quenched and Tempered Alloy Steel Bolts and Studs with Suitable Nuts (A 354 - 62 T)

Spec. for Electric-Fusion-Welded Austenitic Chromium-Nickel Alloy Steel Pipe for High-Temperature Service (A 358 - 62 T)

Spec. for Carbon and Alloy Steel Forgings for Pressure Vessel Shells (A 372 - 62 T)

Spec. for Seamless Austenitic Steel Pipe for High-Temperature Central-Station Service (A 376 - 62 T)

Spec. for Galvanized Steel Transmission Tower Bolts and Nuts (A 394 - 61 T)

Spec. for Seamless Ferritic Alloy-Steel Pipe Specially Heat-Treated for High-Temperature Service (A 405 - 62 T)

Spec. for Upholstery Spring Wire for Coiled Type Springs (A 407 - 57 T)

Spec. for Welded Large Outside Diameter Light-Wall Austenitic Chromium-Nickel Alloy Steel Pipe for Corrosive or High-Temperature Service (A 409 - 60 T)

Spec. for Upholstery Spring Wire for Zig-Zag Type and Square Formed Type and No-Sag Type Spring Units (A 417 - 57 T)

Spec. for Factory-Made Wrought Carbon Steel and Alloy Steel Welded Fittings of Seamless or Welded Construction for Low-Temperature Service (A 420 - 61 T)

Spec. for Centrifugally Cast Ferritic Alloy Steel Pipe for High-Temperature Service (A 426 - 62 T)

Spec. for General Requirements for Carbon, Ferritic Alloy, and Austenitic Alloy Steel Tubes (A 450 - 62 T) (Jointly with Committee A-10)

Spec. for Centrifugally Cast Austenitic Steel Pipe for High-Temperature Service (A 451 - 62 T) (Jointly with Committee A-10)

Spec. for Centrifugally Cast Austenitic Cold-Wrought Pipe for High-Temperature Service (A 452 - 62 T) (Jointly with Committee A-10)

Method for Ultrasonic Testing and Inspection of Turbine and Generator Steel Rotor Forgings (A 418 - 57 T)

Spec. for Hard-Drawn Steel Spring Wire (A 227 - 47) (Standard reverted to tentative)

Spec. for Steel Music Spring Wire (A 228 - 51) (Standard reverted to tentative)

Spec. for Oil-Tempered Steel Spring Wire (A 229 - 56) (Standard reverted to tentative)

Spec. for Carbon-Steel Valve Spring Quality Wire (A 230 - 47) (Standard reverted to tentative)

Spec. for Chromium-Vanadium Steel Spring Wire (A 231 - 41) (Standard reverted to tentative)

Spec. for Chromium-Vanadium Steel Valve Spring Quality Wire (A 232 - 47) (Standard reverted to tentative)

Spec. for Chromium-Silicon Steel Spring Wire (A 401 - 58) (Standard reverted to tentative)

The revision of the following tentative specifications, consisting of the addition of a new Section 3 on Basis of Purchase, was inadvertently listed in the preprinted report under Editorial Changes:

Spec. for Seamless Carbon Steel Boiler Tubes (A 83 - 62 T)

Spec. for Seamless Low-Carbon and Carbon-Molybdenum Steel Still Tubes for Refinery Service (A 161 - 62 T)

Spec. for Electric-Resistance-Welded Carbon Steel Boiler Tubes (A 178 - 62 T)

Spec. for Seamless Cold-Drawn Low-Carbon Steel Heat-Exchanger and Condenser Tubes (A 179 - 62 T)

Spec. for Seamless Carbon Steel Boiler Tubes for High-Pressure Service (A 192 - 62 T)

Spec. for Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes (A 199 - 62 T)

Spec. for Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service (A 200 - 62 T)

Spec. for Seamless Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 209 - 62 T)

Spec. for Seamless Medium-Carbon Steel Boiler and Superheater Tubes (A 210 - 62 T)

Spec. for Seamless Ferritic and Austenitic Alloy-Steel Boiler, Superheater, and Heat Exchanger Tubes (A 213 - 62 T)

Spec. for Electric-Resistance-Welded Carbon Steel Heat Exchanger and Condenser Tubes (A 214 - 62 T)

Spec. for Electric-Resistance-Welded Carbon Steel Boiler and Superheater Tubes for High-Pressure Service (A 226 – 62 T)
Spec. for Welded Austenitic Steel Boiler, Superheater, Heat Exchanger, and Condenser Tubes (A 249 – 62 T)
Spec. for Electric-Resistance-Welded Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 250 – 62 T)
Spec. for Seamless and Welded Ferritic Stainless Steel Tubing for General Service (A 268 – 62 T) (Jointly with Committee A-10)
Spec. for Seamless and Welded Austenitic Stainless Steel Tubing for General Service (A 269 – 62 T) (Jointly with Committee A-10)
Spec. for Seamless and Welded Austenitic Stainless Steel Sanitary Tubing (A 270 – 62 T) (Jointly with Committee A-10)
Spec. for Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 – 62 T) (Jointly with Committee A-10)
Spec. for Ferritic Alloy Steel Forged and Bored Pipe for High-Temperature Service (A 369 – 62 T)
Spec. for Seamless and Electric Welded Low Alloy Steel Tubes (A 423 – 62 T)
Spec. for Austenitic Steel Forged and Bored Pipe for High-Temperature Service (A 430 – 62 T)

Adopted as Standard:

Spec. for High Hardenability Bearing Steels (A 485 – 63)

Adopted as Standard, Revisions in:

Spec. for Low-Carbon Steel Joint Bars (A 3 – 54)
Spec. for Medium-Carbon Steel Joint Bars (A 4 – 54)
Spec. for Electric-Fusion (Arc)-Welded Steel Plate Pipe (Sizes 16 in. and Over) (A 134 – 62)
Spec. for Electric-Fusion (Arc)-Welded Steel Pipe (Sizes 4 in. and Over) (A 139 – 62)
Spec. for Spiral-Welded Steel or Iron Pipe (A 211 – 54)
Spec. for Metal-Arc Welded Steel Pipe for High-Pressure Transmission Service (A 381 – 61)

Committee A-5 on Corrosion of Iron and Steel:

Report presented by C. W. Straitor, Jr., chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Aluminum-Coated Steel Chain-Link Fence Fabric (A 491 – 63 T)

Accepted as Tentative, Revisions in:

Spec. for Zinc-Coated Steel Chain-Link Fence Fabric (A 392 – 59 T)

Adopted as Standard:

Spec. for Zinc-Coated Flat Steel Armoring Tape (A 459 – 61 T)

Adopted as Standard, Revisions in:

Spec. for Zinc (Hot-Galvanized) Coatings on Products Fabricated from Rolled, Pressed, and Forged Steel Shapes, Plates, Bars, and Strip (A 123 – 59)

Committee A-6 on Magnetic Properties:

Report presented by A. C. Beiler, chairman, and accepted as a report of progress.

Committee B-1 on Wires for Electrical Conductors:

Report presented by D. Halloran, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Aluminum Alloy 5005-H19 Wire for Electrical Purposes (B 396 – 63 T)
Spec. for Concentric-Lay-Stranded 5005-H19 Aluminum Alloy Conductors (B 397 – 63 T)
Spec. for Aluminum Alloy 6201-T81 Wire for Electrical Purposes (B 398 – 63 T)
Spec. for Concentric-Lay-Stranded 6201-T81 Aluminum Alloy Conductors (B 399 – 63 T)
Spec. for Compact Round Concentric-Lay-Stranded ED Grade Aluminum Conductors, Hard-Drawn (B 400 – 63 T)
Spec. for Compact Round Concentric-Lay-Stranded Aluminum Conductors, Steel-Reinforced (ACSR) (B 401 – 63 T)

Accepted as Tentative, Revisions in:

Spec. for Concentric-Lay-Stranded Aluminum Conductors, Steel-Reinforced (ACSR) (B 232 – 62 T)
Spec. for Aluminum-Coated (Aluminized) Steel Core Wire for Aluminum Conductors, Steel Reinforced (ACSR) (B 341 – 62 T)
Def. of Terms Relating to Uninsulated Metallic Electrical Conductors (B 354 – 62 T)
Spec. for Nickel-Coated Soft or Annealed Copper Wire (B 355 – 62 T)

Adopted as Standard:

Test for Electrical Conductivity by Use of Eddy Currents (B 342 – 61 T)

Adopted as Standard, Revisions in:

Spec. for Soft or Annealed Copper Wire (B 3 – 56)

Spec. for Bronze Trolley Wire (B 9 – 55)
Spec. for Tinned Soft or Annealed Copper Wire for Electrical Purposes (B 33 – 58)
Spec. for Soft Rectangular and Square Bare Copper Wire for Electrical Conductors (B 48 – 58)
Spec. for Lead-Coated and Lead-Alloy-Coated Soft Copper Wire for Electrical Purposes (B 189 – 58)
Spec. for Concentric-Lay-Stranded Aluminum Conductors, Hard, Three-Quarter Hard, and Half-Hard (B 231 – 60)
Spec. for Standard Weight Zinc-Coated (Galvanized) Steel Core Wire for Aluminum Conductors, Steel Reinforced (ACSR) (B 245 – 62)
Spec. for Zinc-Coated (Galvanized) Steel Core Wire (with Coatings Heavier than Standard Weight) for Aluminum Conductors, Steel Reinforced (ACSR) (B 261 – 62)
Spec. for Silver-Coated Soft or Annealed Copper Wire (B 298 – 62)

There failed of approval at the session the proposed Tentative Specification for Hard-Drawn Aluminum Clad Steel Wire and the proposed Tentative Specification for Concentric - Lay - Stranded Aluminum - Clad Steel Conductors.

Committee B-2 on Nonferrous Metals and Alloys:

Report presented in the absence of the chairman by C. K. Conard, secretary, and accepted as a report of progress.

Committee E-2 on Emission Spectroscopy:

Report presented by R. E. Michaelis, chairman, and the following actions taken subject to a favorable letter ballot return in Committee E-2:

Accepted as Tentative:

Method for Spectrochemical Analysis of Plain-Carbon and Low-Alloy Steel by the Rod-to-Rod Spark Technique (E 212 – 63 T)

Accepted as Tentative, Revisions in:

Def. of Terms and Symbols Relating to Emission Spectroscopy (E 135 – 60 T)
Rec. Practice for Designation of Shapes and Sizes of Preformed Graphite Electrodes (E 130 – 57 T)

Accepted for Publication as Information Only:

Method for Spectrochemical Analysis of Gold-Antimony Alloys by the Solution-Residue Graphite-Spark Technique
Method for Spectrochemical Analysis of Lead and Its Alloys by the Point-to-Plane, Argon Atmosphere, Spark Technique Using an Optical Emission Spectrometer
Method for Spectrochemical Analysis of Gold Alloys by the Solution-Rotating Disk-Spark Technique
Method for Spectrochemical Analysis of Indium and Its Alloys by the Point-to-Plane Condensed Arc Technique Using an Optical Emission Spectrometer
Method for Spectrochemical Analysis of Gallium-Lead and Antimony-Lead Alloys by the Solution-Porous Cup-Spark Technique
Method for Spectrochemical Analysis of Silver by the Powder - D-C Arc Technique
Method for Spectrochemical Analysis of Stainless-Type Steels by the Point-to-Plane Spark Technique Using an Optical Emission Spectrometer
Method for Spectrochemical Analysis of Stainless-Type 18/8 Steels by the Point-to-Plane Technique Using an Optical Emission Spectrometer
Method for Spectrochemical Analysis of Cement Raw Mix by the Lithium Tetraborate Fusion Technique Using a Recording X-ray Spectrometer
Revision of Suggested Method for Spectrochemical Analysis of Zirconium by the Silver Chloride Carrier - D-C Arc Technique

Committee E-3 on Chemical Analysis of Metals:

Report presented by Arba Thomas, chairman, and the following actions taken subject to a favorable letter ballot vote in Committee E-3:

Accepted as Tentative, Revisions in:

Methods for Chemical Analysis of Zirconium and Zirconium-Base Alloys (E 146 – 62 T)
Test for Hydrogen Loss of Copper and Tungsten Powders (E 159 – 62 T)
Methods for Chemical Analysis of Reactor and Commercial Columbium (E 195 – 62 T)

Adopted as Standard:

Methods for Chemical Analysis of Ferro-Alloys (E 31 – 60 T)
Methods for Chemical Analysis of Magnesium and Magnesium-Base Alloys (E 35 – 60 T)
Methods for Chemical Analysis of Silver Brazing Alloys (E 56 – 60 T)
Methods for Chemical Analysis of Titanium and Titanium-Base Alloys (E 120 – 60 T)

Committee E-4 on Metallography:

Report presented by L. L. Wyman, chairman, and the following actions taken:

Adopted as Standard:

Method for Preparing Quantitative Pole Figures of Metals (E 81 - 54 T)
Method for Determining the Orientation of a Metal Crystal (E 82 - 49 T)
Method for Assigning Phase Designations in Metallic Systems (E 157 - 61 T)
Def. of Terms Relating to Metallography (E 7 - 55 T)
Rec. Practice for Thermal Analysis of Metals and Alloys (E 14 - 51 T)
Rec. Practice for Determining the Inclusion Content of Steel (E 45 - 62 T)
Rec. Practice for Dilatometric Analysis of Metallic Materials (E 80 - 49 T)
Rec. Practice for Resistometric (or EMF) Analysis of Metallic Materials (E 189 - 61 T)

Adopted as Standard, Revisions in:

Methods for Estimating the Average Grain Size of Metals (E 112 - 61)

Joint Committee on Effect of Temperature on the Properties of Metals:

Report presented by G. V. Smith, chairman, and accepted as a report of progress.

MARBURG LECTURE[12]

WEDNESDAY, JUNE 26, 5:00 P.M.

SESSION CHAIRMAN: A. C. WEBBER

A brief statement of the purpose of the lecture was given by President Webber. R. F. Blomquist, Research Chemist, U. S. Forest Products Laboratory, was then introduced by President Webber as the thirty-seventh Edgar Marburg Lecturer. Speaking on the subject "Adhesives—Past, Present and Future," Mr. Blomquist traced the development of the art of sticking things together from the time of the early Egyptians to the present. He reviewed what can now be done with adhesives and speculated on what our new basic knowledge will mean in future developments.

President Webber expressed appreciation to Mr. Blomquist for his excellent lecture and presented to him on behalf of the Society the Edgar Marburg Lecture Certificate.

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[12] To be issued as a separate publication.

COCKTAIL PARTY AND ANNUAL DINNER

WEDNESDAY, JUNE 26, 6:30 P.M.

The Philadelphia District through its council acted as the official host at the cocktail party and dinner which was followed by entertainment and dancing.

SYMPOSIUM ON RECENT ADVANCES IN ADHESIVES TECHNOLOGY[10]

(*Sponsored by Committee D-14 on Adhesives*)

THURSDAY, JUNE 27, 9:00 A.M.

SESSION CHAIRMAN: R. F. BLOMQUIST

Physical Properties of Various Epoxy Formulations—N. J. DeLollis, presented by the author.

Phenoxy Resin—A New Thermoplastic Adhesive—T. E. Bugel, S. Norwalk, and R. H. Snedeker, presented by Mr. Bugel.

Adhesive Requirements in the Automotive Industry—S. B. Twiss, presented by the author.

The Relative Efficiency of Phenolics and Ureas in Particle Board Binding—R. H. Kamrath, presented by the author.

Capsular Adhesives—J. D. Bonfiglio, presented by the author.

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[10] Issued with the Symposium on Recent Developments in Adhesion Science as *ASTM STP 360.*

(*Continued at 2:30 p.m.*)

SYMPOSIUM ON X-RAY AND ELECTRON PROBE ANALYSIS[13]

(*Sponsored jointly by Committees E-2 on Emission Spectroscopy and E-4 on Metallography*)

THURSDAY, JUNE 27, 9:00 A.M.

SESSION CHAIRMAN: R. E. MICHAELIS

Production of X-rays in X-ray Emission Spectrography—P. D. Zemany, presented by the author.

X-Ray Optics in Electron Microanalysis—R. E. Ogilvie, presented by the author.

X-ray Detectors and Counting Techniques—W. R. Kiley and J. A. Dunne, presented by Mr. Kiley.

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[13] Issued as *ASTM STP 349.*

(*Continued at 2:00 p.m. and 8:00 p.m. and on Friday, June 28, at 9:00 a.m.*)

SYMPOSIUM ON SPACE RADIATION EFFECTS[9] (*Cont.*)

THURSDAY, JUNE 27, 9:00 A.M.

CO-CHAIRMEN: W. R. LUCAS
J. F. WATSON

Forum: Nuclear Radiation Effects on Materials for Space Application

Effects of Vacuum and Nuclear Radiation on Engineering Materials—W. R. Lucas and G. C. Marshall, presented by Mr. Lucas.

Temperature-Radiation Effects on Materials in Space: A Review—J. F. Watson, presented by the author.

Panel Members: Short presentations by each panel member:

L. K. Zoller, National Aeronautics and Space Administration

J. Romanko, General Dynamics/Fort Worth

Park H. Miller, General Dynamics/Fort Worth

R. E. Gaumer, Lockheed Aircraft Corp.

Open discussion.

SYMPOSIUM ON RESINOGRAPHIC METHODS[14]

(Sponsored by Subcommittee 28 of Committee E-1 on Methods of Testing)

THURSDAY, JUNE 27, 9:00 A.M.

SESSION CHAIRMAN: T. G. ROCHOW

Introduction—T. G. Rochow, presented by the author.

The Preparation of Resins for Microscopical Examination by Reflected Light—J. I. Gedney and E. J. Thomas, presented by Mr. Gedney.

Transparent Section Microscopy and High-Speed Cinematography in Photoelasticity Studies—R. E. Wright and N. W. Hall, presented by Mr. Wright.

Microscopical Apparatus and Techniques for Observing the Fiber-Forming Process—A. O. Mogensen, presented by the author.

Analysis in Polarized Light of Submicroscopic Structures in Resins—M. Nahmmacher, presented by the author.

(Continued at 2:00 p.m. and 8:00 p.m. and on Friday, June 28, at 9:00 a.m.)

COMMITTEE REPORT SESSION

THURSDAY, JUNE 27, 11:30 A.M.

SESSION CHAIRMAN: G. H. HARNDEN

Committee B-5 on Copper and Copper Alloys, Cast and Wrought:

Report presented by W. H. Jennings, chairman, and the following actions taken:

Adopted as Standard:

Spec. for Copper Nickel Alloy Plate and Sheet for Pressure Vessels (B 402 – 63)
Spec. for Seamless Brass Tube (B 135 – 62 T)
Spec. for Seamless Copper and Copper-Alloy Rectangular Waveguide Tube (B 372 – 62 T)
Spec. for U-Bend Seamless Copper and Copper Alloy Heat Exchanger and Condenser Tubes (B 395 – 62 T)

Adopted as Standard, Revisions in:

Spec. for Steam or Valve Bronze Castings (B 61 – 60)
Spec. for Composition Bronze or Ounce Metal Castings (B 62 – 60)
Spec. for Leaded Red Brass and Leaded Semi-Red Brass Sand Castings (B 145 – 61)
Spec. for High-Strength Yellow Brass (Manganese Bronze) and Leaded High-Strength Yellow Brass (Leaded Manganese Bronze) Sand Castings (B 147 – 52)
Spec. for Copper-Nickel-Zinc Alloy (Nickel Silver) Rod and Bar (B 151 – 58)

Committee C-11 on Gypsum:

Report presented by G. W. Josephson, chairman, and the following actions taken:

Accepted as Tentative:

Specs. for Nails for the Application of Gypsum Wallboard (C 514 – 63 T)

Adopted as Standard:

Spec. for Precast Reinforced Gypsum Slabs (C 377 – 58 T)
Spec. for Annular Ringed Nails for Gypsum Wallboard (C 380 – 61 T)
Spec. for Gypsum Backing Board (C 442 – 61 T)

Adopted as Standard, Revisions in:

Spec. for Gypsum Plasters (C 28 – 60)

Committee D-10 on Packaging:

Report presented by J. W. Goff, chairman, and accepted as a report of progress subject to a favorable letter ballot return in Committee D-10.

Committee D-12 on Soaps and Other Detergents:

Report presented by W. Stericker, chairman, and the following actions taken:

Accepted as Tentative:

Specs. for Combination Bar Soap (D 2179 – 63 T)
Test for Active Oxygen in Bleaching Compounds (D 2180 – 63 T)

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[14] Issued as *ASTM STP 348.*

Adopted as Standard:

Methods of Sampling and Chemical Analysis of Alkylbenzene Sulfonates (D 1568 – 60 T)
Methods of Sampling and Chemical Analysis of Fatty Alkyl Sulfates (D 1570 – 60 T)

Accepted for Publication as Information Only:

Spec. for Drycleaning Detergent, Non-Charge Type
Simplified Ion Exchange Method for Analysis of Sodium Triphosphate
Test for Sodium Carboxymethyl Cellulose (CMC) in Detergent Mixtures

Committee D-13 on Textile Materials:

Report presented in the absence of the chairman by Josephine M. Blandford, and the following actions taken:

Accepted as Tentative:

Test for Seed Coat Fragments and Funiculi in Cotton Fiber Samples (D 2496 – 63 T)
Tolerances for Man-Made Organic Base Filament Single Yarns (D 2497 – 63 T)

Accepted as Tentative, Revisions in:

Test for Length of Cotton Fibers by Fibrograph (D 1447 – 54 T)
Test for Linear Density of Textile Fibers by the Vibroscope (D 1577 – 60 T)
Def. of Terms Relating to Textile Materials (D 123 – 62)
Test for Tensile Strength of Wool Fiber Bundles (D 1294 – 58) (Standard reverted to tentative)

Adopted as Standard:

Def. of Terms Relating to Zippers (D 2050 – 61 T)
Test for Durability of Finish of Zippers to Laundering (D 2051 – 61 T), revised as follows:

Section 2.—Change to read as follows:

2. The apparatus and materials shall be as specified in AATCC Method 36 - 1961, Colorfastness to Washing; Characterization of Textile Colorants.[1]

Section 5.—Change the reference to AATCC Method 61 - 1961 T to "Method 36 - 1961."

Test for Colorfastness of Zippers to Drycleaning (D 2052 – 61 T)
Test for Colorfastness of Zippers to Light (D 2053 – 61 T)
Test for Colorfastness of Zipper Tapes to Crocking (D 2054 – 61 T)
Test for Colorfastness of Zipper Tapes to Perspiration (D 2055 – 61 T)
Test for Resistance of Finish of Zippers to Dry Abrasion (D 2056 – 61 T)
Test for Colorfastness of Zippers to Laundering (D 2057 – 61 T), revised as follows:

Section 2.—Change to read as follows:

2. The apparatus shall be as specified in AATCC Method 36 - 1961, Colorfastness to Washing; Characterization of Textile Colorants.[1]

Section 5.—Change the reference to AATCC Method 61 - 1961 T to "Method 36 - 1961."

Test for Durability of Finish of Zippers to Drycleaning (D 2058 – 61 T)
Test for Resistance of Zippers to Salt Fog (D 2059 – 61 T)
Tests for Measuring Zippers (D 2060 – 61 T)
Methods for Strength Tests of Zippers (D 2061 – 61 T)
Test for Operability of Zippers (D 2062 – 61 T)
Test for Predicting Differential Dyeing Behavior of Cotton (D 1464 – 57 T)

Adopted as Standard, Revisions in:

Def. of Terms Relating to Textile Materials (D 123 – 62), with the deletion of Note 1 to the definition of Tenacity, and the deletion of the definitions for Modulus, Secant; and Strength, Tensile, True.
Spec. for Fineness (Grade) of Wool Tops (D 472 – 61)
Methods of Testing Nonwoven Fabrics (D 1117 – 62)
Tests for Water Resistance of Textile Fabrics (D 583 – 58)
Test for Tear Resistance of Woven Fabrics by Falling Pendulum (Elmendorf) Apparatus (D 1424 – 59)
Test for Cross-Sectional Characteristics of Cotton Fibers (D 1444 – 56)

Standards Withdrawn:

Tolerances for Filament Acetate Yarns (D 1235 – 58)
Tolerances for Filament Nylon Yarns (D 1236 – 58)
Tolerances for Filament Rayon Yarns (D 1237 – 58)
Test for Stretch of Hosiery (D 1058 – 51)

Tentative Withdrawn:

Test for Snag Resistance of Hosiery (D 1115 – 54 T)

The session requested that the reason for the withdrawal of these standards and tentative be included in the D-13 Report when the report is published in the *Proceedings*.

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[1] 1961 *Technical Manual*, Am. Assn. Textile Chemists and Colorists.

Committee D-14 on Adhesives:

No action was taken at the Annual Meeting on the Report of Committee D-14 in the absence of an individual authorized to present the report. Alternate procedures will be used in securing Society action on the recommendations in the report as follows:

The recommendations relating to tentatives will be submitted to the Administrative Committee on Standards for action on behalf of the Society.

The revision for immediate adoption of the Standard Methods of Test for Consistency of Adhesives (D 1084 - 60) will be handled under the newly instituted "Interim Procedure" for standards. This revision has been placed on the Society Letter Ballot so that members will have an opportunity to vote on the revision. The results of the ballot on this item are then subject to review by the Administrative Committee on Standards together with the letter ballot vote in the committee. The vote in Committee D-14 on the revision of Methods D 1084 - 60 resulted as follows: From a membership of 143, 82 members returned ballots, of whom 65 voted affirmatively, 0 negatively, and 17 members marked their ballots "not voting."

Committee D-15 on Engine Antifreezes:

No action was taken at the Annual Meeting on the Report of Committee D-15 in the absence of a quorum at the session when the report was scheduled for presentation. All recommendations in the report relate to tentatives and they will be submitted to the Administrative Committee on Standards for action on behalf of the Society.

Committee D-17 on Naval Stores:

No action was taken at the Annual Meeting on the Report of Committee D-17 in the absence of a quorum at the session when the report was scheduled for presentation. The two recommendations in the report—the adoption as standard with revision of Tentative Methods of Test for Fatty Acids Content of Tall Oil Rosin (D 1585 - 58 T) and the revision for immediate adoption of Standard Definitions of Terms Relating to Naval Stores and Related Products (D 804 - 57)—will be processed in the Society under the newly instituted "Interim Procedure" for standards. The recommendations have been placed on the Society Letter Ballot so that members will have an opportunity to vote on them. The results of the ballot on these two items are then subject to review by the Administrative Committee on Standards.

The vote in Committee D-17 on the revision and adoption as standard of Tentative Methods D 1585 - 58 T resulted in 16 affirmative votes, 0 negative, and 3 "not voting" ballots. The revision for immediate adoption of Standard Definitions D 804 - 57 received 17 affirmative votes, 0 negative, and 2 "not voting" ballots. There are 23 members of Committee D-17.

Committee D-20 on Plastics:

Report presented by J. E. Hanna, chairman, and the following actions taken:

Accepted as Tentative:

Rec. Practice for Statistical Design in Interlaboratory Testing of Plastics (D 2188 - 63 T), subject to editorial changes as suggested by Committee E-11 on Quality Control of Materials.

Accepted as Tentative, Revisions in:

Spec. for Polyethylene Molding and Extrusion Materials (D 1248 - 60 T)
Spec. for Polytetrafluoroethylene Rod (D 1710 - 60 T)

Adopted as Standard:

Spec. for Cellulose Acetate Molding and Extrusion Compounds (D 706 - 61 T)
Spec. for Cellulose Acetate Butyrate Molding and Extrusion Compounds (D 707 - 61 T)
Spec. for Ethyl Cellulose Molding and Extrusion Compounds (D 787 - 61 T)
Spec. for Methacrylate Molding and Extrusion Compounds (D 788 - 63 T)
Spec. for Biaxially Oriented Styrene Plastics Sheet (D 1463 - 58 T)
Test for Stiffness of Plastics by Means of a Cantilever Beam (D 747 - 61 T)
Test for Coefficients of Friction of Plastic Film (D 1894 - 61 T)
Test for Absorbed Gamma Radiation Dose in the Fricke Dosimeter (D 1671 - 59 T) (Jointly with Committee D-9)
Test for Water Absorption of Plastics (D 570 - 59a T)
Test for Particle Size (Sieve Analysis) of Plastic Materials (D 1921 - 61 T)
Test for Measuring Flatness of Plastics Sheet or Tubing (D 1604 - 58 T)
Test for Apparent Density of Rigid Cellular Plastics (D 1622 - 59 T)
Rec. Practice for Determining Resistance of Plastics to Fungi (D 1924 - 61 T)
Rec. Practice for Injection Molding of Speci-

mens of Thermoplastic Materials (D 1130 - 50 T)
Test for Flammability of Rigid Plastics over 0.050 in. in Thickness (D 635 - 56 T)

The committee withdrew from the report the recommendations for adoption as standard of Tentative Specification D 1710 - 60 T, Tentative Methods D 1526 - 58 T, D 1822 - 61 T, D 1893 - 61 T, and D 1623 - 59 T, and Tentative Recommended Practices D 1499 - 59 T.

Adopted as Standard, Revisions in:

Test for Flexural Properties of Plastics (D 790 - 61)
Test for Gas Transmission Rate of Plastic Sheeting (D 1434 - 58)

Standard Withdrawn:

Spec. for Vinyl Chloride-Acetate Resin Molding Compounds (D 728 - 50)

Tentative Withdrawn:

Spec. for Vinyl Chloride Polymer and Copolymer Rigid Sheets (D 708 - 53 T)

Committee D-23 on Cellulose and Cellulose Derivatives:

No action was taken at the Annual Meeting on the Report of Committee D-23 in the absence of a quorum at the session when the report was scheduled for presentation. Alternate procedures will be used in securing Society action on the recommendations in the report as follows:

The revision of Tentative Methods of Testing Sodium Carboxymethylcellulose (D 1439 - 61 T) will be submitted to the Administrative Committee on Standards for action on behalf of the Society.

The other three recommendations—the adoption as standard of Tentative Methods of Test for Chromatographic Analysis of Chemically Refined Cellulose (D 1915 - 61 T) and Carboxyl Content of Cellulose (D 1926 - 61 T), and Tentative Methods of Testing Cellulose Acetate (D 871 - 61 T)—will be handled under the newly instituted "Interim Procedure" for standards. These recommendations have been placed on the Society Letter Ballot so that members will have an opportunity to vote on them. The results of the ballot on these three items are then subject to review by the Administrative Committee on Standards together with the letter ballot vote in the committee.

The vote in Committee D-23 on these three recommendations resulted as follows: From a membership of 46, 32 members returned ballots, of whom 25 voted affirmatively, 0 negatively, and 7 members marked their ballots "not voting."

Committee E-12 on Appearance:

Report presented by G. W. Ingle, chairman, and the following actions taken:

Accepted as Tentative:

Def. of Terms Relating to Carbon Paper and Inked Ribbon Products and Images Made Therefrom (E 221 - 63 T)

Adopted as Standard:

Rec. Practice for Goniophotometry of Transmitting Objects and Materials (E 166 - 60 T)
Rec. Practice for Goniophotometry of Reflecting Objects and Materials (E 167 - 60 T)

Committee F-2 on Flexible Barrier Materials:

Report presented in the absence of the chairman by J. W. Goff and accepted as a report of progress subject to a favorable letter ballot return in Committee F-2.

SYMPOSIUM ON RESINOGRAPHIC METHODS[14] *(Cont.)*

THURSDAY, JUNE 27, 2:00 P.M.

SESSION CHAIRMAN: F. F. MOREHEAD

Surface Properties of Fibers—S. C. Scheier and W. J. Lyons, presented by Mr. Scheier.
Light Scattering of Filaments—C. K. Sloan, presented by Mr. Sloan.
Light Scattering Studies of the Internal Structure of Films and Fibers—M. B. Rhodes and R. S. Stein, presented by Mr. Stein.
The Electron Microscope Applied to a Study of Polyethylene Fibers—R. G. Scott, presented by the author.

(Continued at 8:00 p.m. and on Friday, June 28, at 9:00 a.m.)

SYMPOSIUM ON X-RAY AND ELECTRON PROBE ANALYSIS[13] (*Cont.*)

THURSDAY, JUNE 27, 2:00 P.M.

SESSION CHAIRMAN: R. E. MICHAELIS

X-ray Spectrographic Analysis of Metals and Minerals: Scope and Limitations of Quantitative Analysis—J. W. Kemp, presented by H. W. Calkins.

Fluorescent X-ray Spectrographic Analysis of Trace Elements Including Thin Films—W. J. Campbell, presented by the author.

X-ray Spectrographic Analysis of Liquids and Solutions—E. L. Gunn, presented by the author.

(Continued at 8:00 p.m. and on Friday, June 28, at 9:00 a.m.)

SYMPOSIUM ON RECENT ADVANCES IN ADHESIVES TECHNOLOGY[10] (*Cont.*)

THURSDAY, JUNE 27, 2:30 P.M.

SESSION CHAIRMAN: N. J. DELOLLIS

"Polyken" Probe Tack Tester—F. H. Hammon, Jr., presented by the author.

Evidence Indicating the Need for an ASTM Standard of Wood Failure—P. L. Northcott, W. V. Hancock, and H. G. M. Colbeck, presented by Mr. Northcott.

Adhesion of Urethane Elastomers—C. F. Blaich, Jr., presented by the author.

An Adhesive-Metal Bonding Technology Advancement—Bonding of the X-21 Wing—H. F. Click, presented by the author.

SYMPOSIUM ON X-RAY AND ELECTRON PROBE ANALYSIS[13] (*Cont.*)

THURSDAY, JUNE 27, 8:00 P.M.

SESSION CHAIRMAN: L. L. WYMAN

Electron Optical Design of Electron Probes—R. M. Fisher, presented by the author.

Methods of Quantitative Electron Probe Analysis—D. B. Wittry, presented by the author.

Comparison of X-ray Fluorescence and Electron Probe Methods: Future Trends—L. S. Birks, presented by the author.

(Continued on Friday, June 28, at 9:00 a.m.)

SYMPOSIUM ON RESINOGRAPHIC METHODS[14] (*Cont.*)

THURSDAY, JUNE 27, 8:00 P.M.

SESSION CHAIRMAN: R. G. SCOTT

Phase Microscopy in Resinography—C. D. Felton and F. F. Morehead, presented by Mr. Morehead.

Some Applications of Ultraviolet Microscopy in Resinography—C. D. Felton, presented by the author.

Fluorescence Microscopy of Resins—H. W. Zieler, presented by the author.

Dispersion Staining as Applied to Organic Polymer Identification—W. C. McCrone, presented by the author.

(Continued on Friday, June 28, at 9:00 a.m.)

SESSION ON CEMENT

(*Sponsored by Committee C-1 on Cement*)

THURSDAY, JUNE 27, 8:00 P.M.

SESSION CHAIRMAN: W. C. HANSEN

Growing Crystals as Sources of Expansion in Portland Cement Concrete—W. C. Hansen, presented by the author.

Control of False Set by the Use of Anhydrite and Gypsum Blends—J. L. Sawyer, presented by the author.

The Use of X-ray for the Analysis of Cement, Raw Materials, and Raw Mix—O. E. Brown, presented by the author.

SESSION ON ROAD AND PAVING MATERIALS

THURSDAY, JUNE 27, 8:00 P.M.

SESSION CHAIRMAN: H. M. SMITH

Correlation of Composition with Durability and Rheology of Asphalts—R. N. Traxler, presented by the author.

A Falling Plunger Viscometer for Determining Asphalt Viscosity at Low Temperatures—L. E. Santucci and R. J. Schmidt, presented by T. K. Jones.

The Rheological Properties of Asphalt-Aggregate Compositions—E. M. Krokosky, E. Tons, and R. D. Andrews, presented by Mr. Krokosky.

Evaluation of Laboratory Techniques of Soaking Soil-Asphalt Specimens—M. Herrin, P. Manke, and Z. George, presented by Mr. Manke.

(*Continued on Friday, June 28, at 9:00 a.m.*)

QUALITY CONTROL AS RELATED TO TEST RESULTS AND SPECIFICATION REQUIREMENTS OF ROAD AND PAVING MATERIALS[15]

(*Sponsored by Committee D-4 on Road and Paving Materials*)

FRIDAY, JUNE 28, 9:00 A.M.

SESSION CHAIRMAN: J. O. IZATT

Establishing Specification Limits by Statistical Data Analysis—C. A. Carpenter, presented by the author.

Significance of Test Results Obtained from Random Samples—J. F. Shook, presented by the author.

Tolerances and Variations of Highway Materials from Specification Limits—W. B. War- and L. D. Sandvig, presented by Mr. Warden.

Uniformity of Asphalt Concrete Plant Production—M. D. Graham, W. C. Burnett, and J. J. Thomas, presented by Mr. Burnett.

SYMPOSIUM ON X-RAY AND ELECTRON PROBE ANALYSIS[13] (*Cont.*)

FRIDAY, JUNE 28, 9:00 A.M.

SESSION CHAIRMAN: L. L. WYMAN

Electron Probe Analysis in Metallurgical Research—K. F. J. Heinrich, presented by the author.

Electron Probe Analysis of Minerals—I. Adler, presented by the author.

Electron Probe Applications to Biology—A. J. Tousimis, presented by the author.

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[15] Issued as *ASTM STP 362.*

SYMPOSIUM ON RESINOGRAPHIC METHODS[14] (*Cont.*)

FRIDAY, JUNE 28, 9:00 A.M.

SESSION CHAIRMAN: R. S. STEIN

Microscopical Techniques for the Study of Solid State Polymerization—D. G. Grabar and C. S. Hsia Chen, presented by Mr. Grabar.

Resinography with 8-A X-rays—S. B. Newman, presented by the author.

Electron Resinography of Polyphase Solids—M. C. Botty, presented by the author.

Microscopical Studies of Heterogeneous Polymeric Systems—G. C. Claver, presented by the author.

Stress Crazing Failure—A Mechanism of Surface Formation Through Wetting—R. E. Coulehan, presented by the author.

COMMITTEE REPORT SESSION

FRIDAY, JUNE 28, 11:30 A.M.

SESSION CHAIRMAN: R. M. BERG

Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys:

Report presented by L. L. Wyman, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Corrosion-Resistant Iron-Chromium and Iron-Chromium-Nickel Alloy Castings for General Application (A 296 - 62 T)

Spec. for Seamless and Welded Austenitic Stainless Steel Pipe (A 312 - 62 T) (Jointly with Committee A-1)

Spec. for Centrifugally Cast Austenitic Steel Pipe for High-Temperature Service (A 451 - 62 T) (Jointly with Committee A-1)

Spec. for Centrifugally Cast Austenitic Cold-Wrought Pipe for High-Temperature Service (A 452 - 62 T) (Jointly with Committee A-1)

The revisions of the following tentative specifications were inadvertently listed in the preprinted report under Editorial Changes:

Spec. for Seamless and Welded Ferritic Stainless Steel Tubing for General Service (A 268 - 62 T) (Jointly with Committee A-1)

Spec. for Seamless and Welded Austenitic Stainless Steel Tubing for General Service (A 269 - 62 T) (Jointly with Committee A-1)

Spec. for Seamless and Welded Austenitic Stainless Steel Sanitary Tubing (A 270 - 62 T) (Jointly with Committee A-1)

Spec. for Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 - 62 T) (Jointly with Committee A-1)

Adopted as Standard:

Spec. for Stainless and Heat Resisting Steel Rope Wire (A 492 - 63)

Spec. for Stainless and Heat Resisting Steel Cold Heading Wire (A 493 - 63)

Spec. for Corrosion-Resisting Chromium-Nickel Steel Plate, Sheet, and Strip (A 167 - 61 T)

Spec. for Corrosion-Resisting Chromium Steel Plate, Sheet, and Strip (A 176 - 61 T)

Spec. for Corrosion-Resisting Chromium Steel Clad Plate, Sheet, and Strip (A 263 - 61 T)

Spec. for Corrosion-Resisting Chromium-Nickel Steel Clad Plate, Sheet, and Strip (A 264 - 61 T)

Spec. for Heat-Resistant Iron-Chromium and Iron-Chromium-Nickel Alloy Castings for General Application (A 297 - 60 T)

Spec. for Iron-Chromium and Iron-Chromium-Nickel Alloy Tubular Centrifugal Castings for General Applications (A 362 - 52 T)

Spec. for High-Strength, High-Temperature Bolting Materials with Expansion Coefficients Comparable to Austenitic Steels (A 453 - 61 T)

Spec. for Hot, Hot-Cold-Worked, and Cold-Worked Alloy Steel Plate, Sheet, and Strip for High Strength at Elevated Temperatures (A 457 - 61 T)

Spec. for Hot, Hot-Cold-Worked and Cold-Worked Alloy Steel Bars for High Strength at Elevated Temperatures (A 458 - 61 T)

Spec. for General Requirements for Delivery of Flat Rolled Stainless and Heat Resisting Steels in Form of Plate, Sheet, and Strip (A 480 - 62 T)

Method of Total Immersion Corrosion Test of Stainless Steels (A 279 - 44 T)

Rec. Practice for Boiling Nitric Acid Test for Corrosion-Resisting Steels (A 262 - 55 T)

Rec. Practice for Conducting Acidified Copper Sulfate Test for Intergranular Attack in Austenitic Stainless Steel (A 393 - 55 T)

Spec. for Corrosion-Resisting Chromium and

Chromium-Nickel Steel Plate, Sheet, and Strip for Fusion-Welded Unfired Pressure Vessels (A 240 – 61 T)
Spec. for Corrosion-Resisting Chromium-Nickel-Manganese Steel Plate, Sheet, and Strip (A 412 – 57 T)
Spec. for Hot-Rolled and Cold-Finished Corrosion-Resisting Chromium-Nickel-Manganese Steel Bars (A 429 – 58 T)
Spec. for Stainless and Heat Resisting Steel Forgings (A 473 – 62 T)
Spec. for Hot Worked, Hot-Cold Worked and Cold Worked Alloy Steel Forgings and Forging Billets for High Strength at Elevated Temperatures (A 477 – 62 T)
Spec. for Chromium-Nickel Stainless Steel Weaving Wire (A 478 – 62 T)
Spec. for Stainless and Heat Resisting Steel Bars and Shapes for Use in Boilers and Other Pressure Vessels (A 479 – 62 T)

Adopted as Standard, Revisions in:

Spec. for Hot-Rolled and Cold-Finished Stainless and Heat Resisting Steel Bars (A 276 – 62)
Spec. for Chromium-Nickel Corrosion-Resisting Steel Spring Wire (A 313 – 55)
Spec. for Corrosion-Resisting Steel Billets and Bars for Reforging (A 314 – 58)

Committee B-7 on Light Metals and Alloys, Cast and Wrought:

Report presented by R. A. Harris, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Magnesium-Base Alloy Investment Castings (B 403 – 63 T)
Spec. for Aluminum-Alloy Seamless Condenser and Heat-Exchanger Tubes with Integral Fins (B 404 – 63 T)

Accepted as Tentative, Revisions in:

Spec. for Factory Made Wrought Aluminum and Aluminum-Alloy Welding Fittings (B 361 – 61 T)
Spec. for Aluminum Foil for Capacitors (B 373 – 61 T)

Adopted as Standard:

Spec. for Aluminum-Base Alloy Sand Castings (B 26 – 62 T)
Spec. for Aluminum-Base Alloy Permanent Mold Castings (B 108 – 62 T)

Adopted as Standard, Revisions in:

Spec. for Magnesium-Base Alloy Sand Castings (B 80 – 62)
Spec. for Magnesium-Base Alloy Sheet and Plate (B 90 – 61)
Spec. for Magnesium-Base Alloy Forgings (B 91 – 60)
Spec. for Magnesium-Base Alloy Bars, Rods, and Shapes (B 107 – 61)
Spec. for Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 179 – 62)
Spec. for Magnesium-Base Alloy Permanent Mold Castings (B 199 – 62)
Spec. for Aluminum-Alloy Sheet and Plate (B 209 – 62)
Spec. for Aluminum-Alloy Drawn Seamless Tubes (B 210 – 62)
Spec. for Aluminum-Alloy Bars, Rods, and Wire (B 211 – 62)
Spec. for Magnesium-Base Alloy Extruded Tubes (B 217 – 60)
Spec. for Aluminum-Alloy Extruded Bars, Rods, and Shapes (B 221 – 62)
Spec. for Aluminum-Alloy Drawn Seamless Tubes for Condensers and Heat Exchangers (B 234 – 61)
Spec. for Aluminum Bars for Electrical Purposes (Bus Bars) (B 236 – 61)
Spec. for Aluminum-Alloy Pipe (B 241 – 62)
Spec. for Aluminum-Alloy Die Forgings (B 247 – 61)
Spec. for Aluminum-Alloy Drawn Seamless Coiled Tubes for Special Purpose Applications (B 307 – 62)
Spec. for Aluminum-Alloy Standard Structural Shapes, Rolled or Extruded (B 308 – 62)
Spec. for Aluminum-Alloy Round Welded Tubes (B 313 – 61)
Spec. for Aluminum-Alloy Rivet and Cold Heading Wire and Rods (B 316 – 61)
Spec. for Extruded Aluminum-Alloy Bar, Rod, Pipe, and Structural Shapes for Electrical Purposes (Bus Conductors) (B 317 – 61)
Spec. for Aluminum Alloy Pipe for Gas and Oil Transmission and Distribution Piping Systems (B 345 – 61)

Standards Withdrawn:

Spec. for Aluminum-Alloy Extruded Tubes (B 235 – 62)
Spec. for Type A and Type B Aluminum-Alloy Drawn Annealed Seamless Coiled Tubes (B 318 – 62)

Committee C-15 on Manufactured Masonry Units:

Report presented in the absence of the chairman by W. A. Whittier, ASTM staff, and the following actions taken:

Accepted as Tentative:

Specs. for Chemical-Resistant Ceramic Tower Packings (C 515 - 63 T)

Adopted as Standard, Revisions in:

Spec. for Sewer Brick (Made from Clay or Shale) (C 32 - 58)

Committee C-21 on Ceramic Whitewares and Related Products:

No action was taken at the Annual Meeting on the Report of Committee C-21 in the absence of an individual authorized to present the report. Alternate procedures will be used in securing Society action on the recommendations in the report as follows:

The tentative revision of Standard Definitions of Terms Relating to Ceramic Whitewares and Related Products (C 242 - 60) will be submitted to the Administrative Committee on Standards for action on behalf of the Society.

The recommendation for adoption as standard of Tentative Definitions of Terms Relating to Ceramic Whitewares and Related Products (C 242 - 60 T) will be handled under the newly instituted "Interim Procedure" for standards.

Committee C-24 on Joint Sealants:

Report presented by W. A. Oberdick, chairman, and the following actions taken:

Accepted as Tentative:

Spec. and Methods of Test for Expanded Elastomeric Preformed Gasket and Sealing Material (C 509 - 63 T)

Test for Staining and Color Change of One- or Two-Part Joint Sealants (C 510 - 63 T)

Committee D-4 on Road and Paving Materials:

Report presented by R. E. Bollen, chairman, and the following actions taken:

Accepted as Tentative:

Test for Kinematic Viscosity of Asphalts (D 2170 - 63 T)

Test for Absolute Viscosity of Asphalts (D 2171 - 63 T)

Test for Quantitative Extraction of Bituminous Paving Mixtures (D 2172 - 63 T)

Specs. for Liquid Asphalt (Slow-Curing Type) (D 2026 - 63 T)

Specs. for Cut-Back Asphalt (Medium Curing Type) (D 2027 - 63 T)

Specs. for Cut-Back Asphalt (Rapid Curing Type) (D 2028 - 63 T)

The new Tentative Specifications D 2027 and D 2028 were inadvertently listed in the preprinted report as revisions with reversion to tentative of Standard Specifications D 598 - 46 and D 597 - 46, respectively.

Accepted as Tentative, Revisions :in

Test for Effect of Heat and Air on Asphaltic Materials (Thin-Film Oven Test) (D 1754 - 60 T)

Spec. for Asphalt Cement for Use in Pavement Construction (D 946 - 62 T)

Tests for Resistance to Deformation and Cohesion of Bituminous Mixtures by Means of Hveem Apparatus (D 1560 - 62 T)

Method for Preparation of Test Specimens of Bituminous Mixtures by Means of California Kneading Compactor (D 1561 - 61 T)

Test for Distillation of Cut-Back Asphaltic Products (D 402 - 55)

Methods of Testing Emulsified Asphalts (D 244 - 60)

Methods of Testing Preformed Expansion Joint Fillers for Concrete (Nonextruding and Resilient Types) (D 545 - 49)

Spec. for Emulsified Asphalt (D 977 - 57) (Standard reverted to tentative)

Adopted as Standard:

Spec. for Coarse Aggregate for Bituminous Paving Mixtures (D 692 - 59 T)

Spec. for Fine Aggregate for Bituminous Paving Mixtures (D 1073 - 59 T)

Test for Recovery of Asphalt from Solution by Abson Method (D 1856 - 61 T)

Test for Sieve or Screen Analysis of Fine and Coarse Aggregates (C 136 - 61 T) (Jointly with Committee C-9)

Adopted as Standard, Revisions in:

Def. of Terms Relating to Materials for Roads and Pavements (D 8 - 55)

Test for Abrasion of Graded Coarse Aggregate by Use of the Deval Machine (D 289 - 55)

Accepted for Publication as Information Only:

Test for Stripping of Bitumen-Aggregate Mixtures (Tracer Salt Method)

Committee D-9 on Electrical Insulating Materials:

Report presented by A. H. Scott, chairman, and the following actions taken:

Adopted as Standard:

Test for Absorbed Gamma Radiation Dose in the Fricke Dosimeter (D 1671 - 59 T) (Jointly with Committee D-20)
Test for Thermal Stability of Electrical Insulating Coated Fabrics by Curved Electrodes (Dielectric Breakdown Method) (D 1830 - 61 T)
Spec. for Vulcanized Fibre Sheets, Rods, and Tubes Used for Electrical Insulation (D 710 - 61 T)
Methods of Testing Vulcanized Fibre Used for Electrical Insulation (D 619 - 59 T)
Methods of Testing Vitrified Ceramic Materials for Electrical Applications (D 116 - 61 T)

Adopted as Standard, Revisions in:

Methods of Testing Pasted Mica Used in Electrical Insulation (D 352 - 59)
Methods of Testing Polymerizable Embedding Compounds Used for Electrical Insulation (D 1674 - 61)

Standards Withdrawn:

Tests for Product Uniformity of Phenolic Laminated Sheets (D 634 - 44)
Methods of Testing Steatite Used for Electrical Insulation (D 667 - 44)

The recommendation in the preprinted report to adopt as standard without change the Tentative Method of Test for Thermal Stability of Electrical Insulating Coated Fabrics by Curved Electrodes (Dielectric Breakdown Method) (D 1830 - 61 T) did not receive the necessary letter ballot approval of Committee D-9. The method has been referred back to the subcommittee for further consideration.

Committee D-25 on Casein and Similar Protein Materials:

Report presented in the absence of the chairman by S. F. Etris, ASTM staff, and accepted as a report of progress.

Committee E-7 on Nondestructive Testing:

Report presented by A. Gobus, chairman, and the following actions taken:

Accepted as Tentative:

Method for Ultrasonic Inspection of Metal Pipe and Tubing for Longitudinal Discontinuities (E 213 - 63 T)
Rec. Practice for Immersed Ultrasonic Testing by the Reflection Method, Using Pulsed Longitudinal Waves (E 214 - 63 T)
Rec. Practice for Electromagnetic Testing of Seamless Aluminum Alloy Tube (E 215 - 63 T)
Rec. Practice for Measuring Coating Thickness by Magnetic or Electromagnetic Methods (E 216 - 63 T)

Adopted as Standard:

Method for Dry Powder Magnetic Particle Inspection (E 109 - 57 T)
Method for Wet Magnetic Particle Inspection (E 138 - 58 T)
Methods for Liquid Penetrant Inspection (E 165 - 60 T)
Reference Radiographs for Steel Welds (E 99 - 55 T)
Rec. Practice for Ultrasonic Testing by the Reflectance Method, Using Pulsed Longitudinal Waves Induced by Direct Contact (E 114 - 55 T)
Reference Photographs for Magnetic Particle Indications on Ferrous Castings (E 125 - 56 T)

Committee E-10 on Radioisotopes and Radiation Effects:

Report presented by G. W. Pomeroy, chairman, and the following actions taken:

Accepted as Tentative:

Method for Determination of Uranium by Controlled-Potential Coulometry (E 217 - 63 T)
Method for Radiochemical Determination of Cesium-137 in Aqueous Solutions (Chloroplatinate Method) (E 218 - 63 T)

Test for Atom Per Cent Fission in Uranium Fuel (Radiochemical Method) (E 219 - 63 T)

Adopted as Standard:

Rec. Practice for Measuring Neutron Flux (E 199 - 62 T)
Def. of Terms Relating to Dosimetry (E 170 - 60 T)

Committee E-11 on Quality Control of Materials:

Report presented by C. A. Bicking, chairman, and accepted as a report of progress.

Committee E-17 on Skid Resistance:

Report presented by E. A. Whitehurst, chairman, and accepted as a report of progress.

COMMITTEE REPORT SESSION

FRIDAY, JUNE 28, 12:30 P.M.

SESSION CHAIRMAN: J. B. RATHER, JR.

Committee A-3 on Cast Iron:

Report presented in the absence of the chairman by J. R. Kattus, secretary, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Ferritic Nodular Iron Castings for Valves, Flanges, Pipe Fittings, and Other Piping Components (A 445 - 60 T)

Adopted as Standard:

Spec. for Austenitic Gray Iron Castings (A 436 - 61 T)

Committee C-1 on Cement:

Report presented by R. R. Litehiser, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Moist Cabinets and Rooms Used in the Testing of Hydraulic Cements (C 511 - 63 T)

Accepted as Tentative, Revisions in:

Spec. for Portland Blast-Furnace Slag Cement (C 205 - 62 T)
Spec. for Portland-Pozzolan Cement (C 340 - 62 T)
Test for Flexural Strength of Hydraulic Cement Mortars (C 348 - 61 T)
Spec. for Slag Cement (C 358 - 62 T)
Test for Potential Sulfate Resistance of Portland Cement (C 452 - 60 T)
Spec. for Processing Additions for Use in the Manufacture of Portland Cement (C 465 - 62 T)
Test for Normal Consistency of Hydraulic Cement (C 187 - 58)

Adopted as Standard, Revisions in:

Spec. for Masonry Cement (C 91 - 60)
Test for Compressive Strength of Hydraulic Cement Mortars (Using 2-In. Cube Specimens) (C 109 - 58)
Spec. for Portland Cement (C 150 - 62), with the exception of the proposed revision to delete in Table II all entries pertaining to tensile strength, and the revised Footnote *d*.
Tests for Autoclave Expansion of Portland Cement (C 151 - 60)
Spec. for Air-Entraining Portland Cement (C 175 - 61), with the addition of a new paragraph (*j*) to Section 10 to read, "(*j*) False Set.—Method of Test for False Set of Portland Cement (Paste Method) (ASTM Designation: C 451)."
Test for Tensile Strength of Hydraulic Cement Mortars (C 190 - 59)

Committee D-2 on Petroleum Products and Lubricants:

Report presented by H. M. Smith, chairman, and the following actions taken:

Accepted as Tentative:

Test for Autoignition Temperature of Liquid Petroleum Products (D 2155 - 63 T)
Test for Smoke Density in the Flue Gases from Distillate Fuels (D 2156 - 63 T)
Test for Effect of Air Supply on Smoke Density in Burning Distillate Fuel (D 2157 - 63 T)
Test for Residue in Liquefied Petroleum (LP) Gases (End Point Index Method) (D 2158 - 63 T)
Test for Naphthenes in Saturates Fractions by Refractivity Intercept (D 2159 - 63 T)
Test for Analysis of Liquefied Petroleum (LP) Gases by Gas Chromatography (D 2163 -

63 T) (Jointly with Committee D-3 on Gaseous Fuels)
Test for Water and Sediment in Crude Oils (D 96 - 63 T)
Spec. for Special Duty Propane (D 2154 - 63 T)
Method for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity or to Saybolt Furol Viscosity (D 2161 - 63 T)
Test for Basic Calibration of Master Viscometers and Viscosity Oil Standards (D 2162 - 63 T)
Test for Thermal Stability of Hydraulic Fluids (D 2160 - 63 T)

Accepted as Tentative, Revisions in:

Spec. for Fuel Oils (D 396 - 62 T)
Test for Aniline Point and Mixed Aniline Point of Petroleum Products and Hydrocarbon Solvents (D 611 - 55 T)
Test for Knock Characteristics of Aviation Fuels by the Aviation Method (D 614 - 60 T)
Spec. for Aviation Gasolines (D 910 - 62 T)
Spec. for Aviation Turbine Fuels (D 1655 - 62 T)
Test for Knock Characteristics of Motor Fuels Above 100 Octane Number by the Research Method (D 1656 - 62 T)
Test for Maximum Fluidity Temperature of Residual Fuel Oil (D 1659 - 59 T)
Test for Thermal Stability of Aviation Turbine Fuels (D 1660 - 61 T)
Test for Knock Characteristics of Motor Fuels Above 100 Octane Number by the Motor Method (D 1948 - 62 T)

Adopted as Standard:

Test for Dilution of Gasoline-Engine Crankcase Oils (D 322 - 58 T)
Test for Ash from Petroleum Oils (D 482 - 59 T)
Test for Chlorine in New and Used Petroleum Products (Bomb Method) (D 808 - 57 T)
Test for Sulfated Ash from Lubricating Oils and Additives (D 874 - 59 T)
Test for Foaming Characteristics of Lubricating Oils (D 892 - 58 T)
Test for Knock Characteristics of Aviation Fuels by the Supercharge Method (D 909 - 60 T)
Test for Water Washout Characteristics of Lubricating Greases (D 1264 - 53 T)
Test for Low-Temperature Torque of Ball Bearing Greases (D 1478 - 57 T)
Test for Vanadium in Navy Special Fuel Oil (D 1548 - 59 T)
Test for Carbon Number Distribution of Aromatic Compounds in Naphthas by Mass Spectrometry (D 1658 - 59 T)
Method for Calculation of Absolute Viscosity (D 1745 - 60 T)
Test for Distillation of Plant Spray Oils (D 447 - 59 T)
Test for Unsulfonated Residue of Petroleum Plant Spray Oils (D 483 - 61 T)

Adopted as Standard, Revisions in:

Test for Melting Point of Petrolatum and Microcrystalline Wax (D 127 - 60)
Test for Knock Characteristics of Motor Fuels of 100 Octane Number and Below by the Motor Method (D 357 - 62)
Test for Knock Characteristics of Motor Fuels of 100 Octane Number and Below by the Research Method (D 908 - 62)

Standards Withdrawn:

Method for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity (D 446 - 53)
Method for Conversion of Kinematic Viscosity to Saybolt Furol Viscosity (D 666 - 57)
Test for Sulfated Residue, Lead, Iron, and Copper in Lubricating Oils (D 810 - 59)
Test for Saponification Number by Potentiometric Titration (D 939 - 54)

Accepted for Publication as Information Only:

Method for Simulative Recirculating System Testing of Aerospace Hydraulic Fluids
Test for Viscosity of a Functional Fluid for Aerospace Application
Field Method for Sampling Aerospace Fluids in Containers
Methods for Sampling Aerospace Liquids in Components
Test for Particulate Contamination of Hydraulic Fluids by Gravimetric Analysis
Test for Rapid Identification of Metallic and Fibrous Contaminants in Aerospace Fluids
Test for Vapor-Liquid Ratio of Gasoline
Test for Interconversion of the Analysis of C_5 and Lighter Hydrocarbons to Gas-Volume, Liquid-Volume, or Weight Basis
Test for Moisture in Liquefied Petroleum (LP) Gases (Valve Freeze Method)
Test for Preliminary Examination of Hydraulic Fluids (Wear Test)
Test for Aromatics in Light Naphthas, Reformates, and Gasolines by Gas Chromatography
Test for Evaluation of White Mineral Oils by Ultraviolet Absorption
Test for Weight of Surface Wax on Waxed Paper
Test for Salt in Crude Oil (Electrometric Method)
Methods for Calculating Viscosity Index

Test for Analysis of Oil-Soluble Petroleum Sulfonates by Liquid Chromatography
Test for Particulate Contaminant in Aviation Fuels
Test for Determining Knock Characteristics of Liquefied Petroleum (LP) Gases by the Motor Method
Method for Detection of Hydrogen Sulfide in Liquefied Petroleum (LP) Gases (Lead Acetate Test)
Test for Vapor Forming Index (VFI) of Motor Gasolines
Test for Kinetic Coefficient of Friction for Wax Coatings
Rapid Combustion Methods for the Determination of Sulfur in Liquid and Gaseous Hydrocarbon Materials (Wickbold and Beckman Burner Techniques)

The committee withdrew from the preprinted report the recommendations for adoption as standard without revision of Tentative Method of Test for Specific Gravity of Light Hydrocarbons by Pressure Hydrometer (D 1657 – 59 T) and the revision for immediate adoption of Standard Method of Test for Kinematic Viscosity (D 445 – 61).

Committee D-3 on Gaseous Fuels:

Report presented in the absence of the chairman by K. R. Knapp, secretary, and the following actions taken:

Accepted as Tentative:

Test for Analysis of Liquefied Petroleum (LP) Gases by Gas Chromatography (D 2163 – 63 T) (Jointly with Committee D-2)

Adopted as Standard, Revisions in:

Tests for Specific Gravity of Gaseous Fuels (D 1070 – 52)
Test for Water Vapor Content of Gaseous Fuels by Measurement of Dew Point Temperature (D 1142 – 58)

Committee D-6 on Paper and Paper Products:

Report presented by H. A. Birdsall, chairman, and the following actions taken:

Accepted as Tentative:

Test for Arsenic in Paper and Paperboard (D 2173 – 63 T)
Test for Tensile Energy Absorption of Paper (D 2174 – 63 T)
Test for Folding Endurance of Paper by the M.I.T. Tester (D 2176 – 63 T)
Test for Ink Absorption of Blotting Paper (D 2177 – 63 T)

Accepted as Tentative, Revisions in:

Test for Fiber Analysis of Paper and Paperboard (D 1030 – 55 T)
Tests for Folding Endurance of Paper (D 643 – 43) (Standard reverted to tentative)
Test for Absorption by Bibulous Papers of Water and Writing Ink (D 824 – 47) (Standard reverted to tentative)
Test for Opacity of Paper and Paper Products (D 589 – 44) (Standard reverted to tentative)

Adopted as Standard, Revisions in:

Test for Machine Direction of Paper (D 528 – 41)
Test for Ash Content of Paper and Paper Products (D 586 – 42)
Tests for Zinc and Cadmium in Paper (D 1224 – 54)

Tentative Withdrawn:

Test for Contrast Gloss of Paper at 57.5 Deg (D 1222 – 52 T)

The committee withdrew from the preprinted report the following recommendations: (1) Proposed Tentative Method of Wax Test for Surface Strength of Paper, (2) Revision of Tentative Methods of Test for Thickness of Paper and Paper Products (D 645 – 58 T), and (3) Withdrawal of Standard Method of Test for Degree of Staining of Paper by Alkali (D 723 – 45).

Committee D-11 on Rubber and Rubber-like Materials:

Report presented by G. C. Maassen, chairman, and the following actions taken:

Adopted as Standard:

Method of Calibrating a Light Source Used for Accelerating the Deterioration of Rubber (D 749 – 43 T)
Spec. for Friction Tape for General Use for Electrical Purposes (D 69 – 59 T)

Adopted as Standard, Revisions in:

Methods of Testing Automotive Air Brake and Vacuum Brake Hose (D 622 – 60)
Tests for Contact and Migration Stain of Vulcanized Rubber in Contact with Organic Finishes (D 925 – 55)
Test for Discoloration of Vulcanized Rubber: Organic Finish Coated or Light Colored (D 1148 – 55)
Test for Viscosity and Curing Characteristics of Rubber by the Shearing Disk Viscometer (D 1646 – 62)

The committee withdrew from the preprinted report the recommendations for adoption as standard of Tentative Method of Test for Resistance to Accelerated Light Aging of Rubber Compounds (D 750 – 55 T) and Tentative Spec. for Rubber Insulating Tape (D 119 – 59 T), and revision for immediate adoption of Standard Method of Measuring Low-Temperature Stiffening of Rubber and Rubber-Like Materials by Means of a Torsional Wire Apparatus (D 1053 – 61).

Committee D-16 on Industrial Aromatic Hydrocarbons and Related Materials:

Report presented by W. E. Sisco, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Method for Calculation of Volume and Weight of Benzene, Toluene, and Paraxylene (D 1555 – 58 T)

Adopted as Standard:

Test for Water in Phenol and Related Materials by the Iodine Reagent Method (D 1631 – 59 T)

Standards Withdrawn:

Spec. for Industrial Grade Toluene (D 842 – 50)
Spec. for Industrial Grade Xylene (D 844 – 50)

Tentative Withdrawn:

Spec. for Industrial 90 Benzene (D 837 – 56 T)

Accepted for Publication as Information Only:

Method for Xylene Isomer Analysis by Gas Chromatography

Committee D-19 on Industrial Water:

Report presented in the absence of the chairman by M. D. Huber, ASTM staff, and the following actions taken:

Accepted as Tentative:

Test for Deuterium Oxide (D 2184 – 63 T)
Test for Deposit-Forming Impurities in Steam (D 2186 – 63 T)
Test for Physical and Chemical Properties of Ion Exchange Resins (D 2187 – 63 T)
Test for Radioactive Cobalt in Industrial Water and Industrial Waste Water (D 2185 – 63 T)

Accepted as Tentative, Revisions in:

Tests for Sulfate Ion in Industrial Water and Industrial Waste Water (D 516 – 59 T)
Tests for Copper in Industrial Water and Industrial Waste Water (D 1688 – 62 T)
Test for Manganese in Industrial Water and Industrial Waste Water (D 858 – 49) (Standard reverted to tentative)
Test for Nitrite Ion in Industrial Water (D 1254 – 60) (Standard reverted to tentative)

Adopted as Standard:

Test for Hexane-Extractable Matter in Industrial Waste Water (D 1891 – 61 T)
Test for Odor in Industrial Water and Industrial Waste Water (D 1292 – 61 T)

Adopted as Standard, Revisions in:

Method of Reporting Results of Analysis of Industrial Water and Industrial Waste Water (D 596 – 58)
Def. of Terms Relating to Industrial Water and Industrial Waste Water (D 1129 – 62)

The committee withdrew from the preprinted report the proposed Tentative Method of Test for Primary and Secondary Amines in Industrial Water.

Committee D-26 on Halogenated Organic Solvents:

Report presented by W. D. McMaster, chairman, and accepted as a report of progress, subject to a favorable letter ballot vote in the committee.

Committee E-15 on Analysis and Testing of Industrial Chemicals:

Report presented by E. G. Wiest, chairman, and the following actions taken:

Accepted as Tentative:

Test for Hydroxyl Groups by Acetic Anhydride Acetylation (E 222 – 63 T)
Methods for Analysis of Sulfuric Acid (E 223 – 63 T)
Methods for Analysis of Hydrochloric Acid (E 224 – 63 T)

Accepted as Tentative, Revisions in:

Rec. Practice for Developing Precision Data on ASTM Methods for Analysis and Testing of Industrial Chemicals (E 180 – 62 T)

Committee E-20 on Temperature Measurement:

No action was taken at the Annual Meeting on the Report of Committee E-20 in the absence of a quorum at the session when the report was scheduled for presentation. Alternate procedures will be used in securing Society action on the recommendations in the report as follows:

The proposed Tentative Method for Comparison Calibration of Thermocouples will be submitted to the Administrative Committee on Standards for action on behalf of the Society.

The revision for immediate adoption of the Standard Specifications for ASTM Thermometers (E 1 – 62) will be handled under the newly instituted "Interim Procedure" for standards. This revision has been placed on the Society Letter Ballot so that members will have an opportunity to vote on the revision. The results of this ballot are then subject to review by the Administrative Committee on Standards together with the letter ballot vote in the committee. The vote in Committee E-20 on the revision of Specifications E 1 – 62 resulted as follows: From a membership of 101, 73 members returned ballots, of whom 41 voted affirmatively, 0 negatively, and 32 members marked their ballots "not voting."

EDITOR'S NOTE.—The recommendations affecting standards referred to letter ballot were approved when the ballot was canvassed on September 30, 1963.

SUMMARY OF PROCEEDINGS OF INTERNATIONAL CONFERENCE ON CREEP

The International Conference on Creep, sponsored by the ASTM-ASME Joint Committee on the Effect of Temperature on Metals, was held under the joint auspices of the ASTM, The American Society of Mechanical Engineers, and the Institution of Mechanical Engineers. The sessions were held in New York City, Aug. 25–29, 1963, to be repeated in London, Sept. 30–Oct. 4, 1963.

The sessions in New York, held at the Biltmore Hotel, were very well attended (298 total registration). The Conference operated under the reporter system in which 77 papers, preprinted in a bound volume, were presented in eight sessions. Each session had two chairmen, one from the United States, the other from Great Britain, and two reporters (both from the United States). Each reporter was responsible generally for half the papers in a session. He presented a 30 to 45 min talk, covering the work and conclusion in each paper, then the subject was open for floor discussion. After this, authors were called upon to answer questions or to speak in rebuttal. The second half of the session operated in the same way. The American session chairman handled the introductions and general announcement while the co-chairman handled the discussion.

The ASTM Lecture on Outstanding Research (Second) was presented on Wednesday morning by Mr. N. P. Allen of the National Physical Laboratory, Teddington, Great Britain. Mr. I. V. Williams, presiding as Chairman, presented the certificate and honorarium to Mr. Allen.

The program was divided in sessions as follows:

1. Fundamental Considerations—Metallurgical Structure, Atomic Lattice, Dislocations.
2. Plasticity Theory—Theoretical and Experimental Studies, Under Uniaxial and Combined Stresses.
3. Thermal or Mechanical Cycling.
4. Criteria for Design—Notch Effects, Tube Bursting, Proof Stress, Creep of Graphite.
5. Creep and Rupture Data of Particular Materials.
6. Relaxation and Application, Interpretation and Extrapolation of Creep and Rupture Data.

A second volume will incorporate the discussion of both the New York meeting and the London conference.

The publications are issued by the Institution of Mechanical Engineers.

SUMMARY OF PROCEEDINGS OF SYMPOSIUM ON LABORATORY SHEAR TESTING OF SOILS

This Symposium on Laboratory Shear Testing of Soils, jointly sponsored by ASTM and the National Research Council of Canada, was held at the National Research Council, Ottawa, Canada on September 9 to 11, 1963.

The following papers and discussions are included in the Symposium:

Strength Testing of Soils—G. F. Sowers

Theories

Shear Strength in a General Stress Field—W. D. L. Finn and H. K. Mittal
The Uniqueness of the Mohr-Coulomb Concept in Shear Failure—E. C. W. A. Geuze
Difference in Triaxial and Plane Strain Shear Strength—Theoretical and Experimental Investigations—H. Leussink and W. Wittke
Creep Studies on Saturated Clays—J. K. Mitchell and R. G. Campanella
A Critical Appreciation of Test Data for Selecting a Yield Criterion for Soils—K. H. Roscoe, A. N. Schofield, and A. Thurairajah
The Stress-Dilatency Performance of Two Clays—P. W. Rowe, D. B. Oates, and N. A. Skermer
Generalizing and Measuring the Effective Components of Shear Resistance—J. H. Schmertmann

Stress Variation and Pore Pressures

The Effect of Nonuniform Pore Pressures on Laboratory Measurements of the Shear Strength of Soils—G. E. Blight
Pore Pressures Within Soil Specimens in Triaxial Compression—C. B. Crawford
Pore Pressure Control and Evaluation for Triaxial Compression—H. J. Gibbs
Pore Pressure Functions in Saturated Soils—E. Juarez-Badillo
Void Ratio Effects on the Hyperbolic Stress-Strain Response of a Sand—R. L. Kondner and J. S. Zelasko
A Graphical Approach to the Problem of the Stress-Strain Relationship of Normally Consolidated Clays—H. B. Poorooshasb and K. H. Roscoe
The Influence of Laboratory Stress Path on Triaxial Test Results—N. Simons
The Influence of Stress History on the Stress Paths Followed in Undrained Triaxial Tests—D. J. Henkel and V. Sowa

Test Methods and New Equipment

The Vienna Method of Shear Testing—H. Borowicka
Triaxial Testing Using Large-Scale High-Pressure Equipment—E. B. Hall and B. B. Gordon
High-Pressure Triaxial Tests on a Compacted Sand and an Undisturbed Silt—R. C. Hirschfeld and S. J. Poulos
The Strength of "Undisturbed" Clay Determined from Undrained Tests—C. C. Ladd and T. W. Lambe
A Simple Pore Water Pressure Gage—G. P. Raymond

Laboratory Vane Shear Tests and the Influence of Pore-Water Stresses—N. E. Wilson
A Transducer to Measure Pore Water Pressures in Soil Tests—K. N. Burn

Test Interpretation and Errors

Influence of Placement Condition and Testing Procedure on Shear Test Results on Compacted Soil—D. J. Bazett and E. S. Bell
The Influence of the Provisions for Boundary Drainage on Strength and Consolidation Measurements in the Triaxial Apparatus—A. W. Bishop and R. E. Gibson
The Controlling Influences of Strain and Creep Phenomena on the Conditions of Shear Failure in Clay Soils—D. M. Burmister
Some Deficiencies in Testing Leda Clay—D. F. Coates and G. C. McRostie
Comparison of the Shear Strengths of Laboratory and Field Compacted Soils—W. G. Holtz and W. Ellis
Effect of Lateral Filter Paper Drains on the Triaxial Shear Characteristics of Soil—R. E. Olson and M. Kiefer
The Effect of Test Techniques on the Shear Strength of Western Canadian Clays—P. J. Rivard and L. Chan

These papers with discussion and reports will be issued as *ASTM Special Technical Publication No. 361* entitled "Symposium on Laboratory Shear Testing of Soils."

SUMMARY OF PROCEEDINGS OF SYMPOSIUM ON STEELS WITH YIELD STRENGTHS OVER 200,000 PSI

A Symposium on Steels with Yield Strengths over 200,000 psi was held in Cleveland, Ohio, at the Statler Hilton Hotel, October 22, 1963 under the co-sponsorship of the Metallurgical Society of the American Institute of Mining, Metallurgical, and Petroleum Engineers and the Panel on Structural Materials for Airframes and Missiles of the ASTM-ASME Joint Committee on Effect of Temperature on the Properties of Metals:

Following is the symposium program:

Relationships Between Microstructure and Toughness in Ultrahigh-Strength Steels—A. J. Baker, F. J. Lauta, and R. P. Wei
Tough, High-Strength Steels for Aerospace and Civilian Applications—S. J. Matas, J. S. Pascover, and H. P. Munger
Anisotropy of Fracture Toughness in High-Strength Plate Materials—R. E. Lewis
An Evaluation of the 18Ni-9Co-5Mo Maraging Steel Sheet—D. L. Corn
The Metallurgy and Properties of AM-355 CRT—A Cold Rolled Semi-Austenitic Stainless Steel—T. H. McCunn, G. N. Aggen, and R. A. Lula
Fracture Micromechanics in High-Strength Steels—B. R. Banerjee
The Effect of Solidification Practice on the Properties of High-Strength Steels—C. M. Carman, D. F. Armiento, and H. Markus
High-Strength Steel Forgings—H. J. Henning
Ausform Fabrication and Properties of High-Strength Alloy Steel—W. W. Gerberich, A. J. Williams, C. F. Martin, and R. E. Heise
Thermomechanical Treatments Applied to Ultrahigh-Strength Bainites—D. Kalish and Morris Cohen
Ultrahigh-Strength Steel Fasteners—A. C. Hood and R. L. Sproat
The Manufacture of Prototype 18 Per Cent Maraging Steel Rocket Casings—E. P. Gilewicz

These papers with discussion will be issued as an ASTM Special Technical Publication entitled "Symposium on Steels with Yield Strengths over 200,000 psi."

SUMMARY OF PROCEEDINGS OF SYMPOSIUM ON CORROSION OF ZIRCONIUM ALLOYS

This Symposium on the Corrosion of Zirconium Alloys, jointly sponsored by the American Nuclear Society and American Society for Testing and Materials, was held at the New York Hilton Hotel, New York, N. Y., on November 21, 1963.

The papers presented were concerned with the problems of corrosion of zirconium and its alloys in nuclear reactors.

The following papers are included in the Symposium:

The Development of the Zircaloys—Stanley Kass
Effect of Fluoride Ions on the Aqueous Corrosion of Zirconium Alloys—Warren E. Berry
Corrosion of Zirconium Alloys by Homogeneous Reactor Fuels—G. H. Jenks
Recent Studies of the Growth and Breakdown of Oxide Films on Zirconium and Zirconium Alloys—J. N. Wanklyn
Effect of Hydrogen and Hydride Morphology on the Tensile Properties of Zircaloy-2—W. J. Babyak, W. F. Bouregeois, and G. J. Salvaggio
The Effect of Water Chemistry and Neutron Irradiation on Corrosion and Hydriding of Zircaloy-2—S. B. Dalgaard
Neutron Irradiation and Cold Work Effects on Zircaloy-2 Corrosion and Hydrogen Pickup—W. A. Burns and H. P. Maffei
Factors Limiting the Utilization of Zirconium Alloys in Superheated Steam—H. H. Klepfer and D. L. Douglass

These papers with discussion will be issued as an ASTM Special Technical Publication entitled "Symposium on Corrosion of Zirconium Alloys."

1963 ANNUAL REPORT OF THE BOARD OF DIRECTORS

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Highlights

Ring Out the Old, Ring in the New! The commencement of construction of the new Society Headquarters building at 1916 Race St. and the demolition of the old buildings to make way for the new symbolized the continuing growth of activity in the Society.

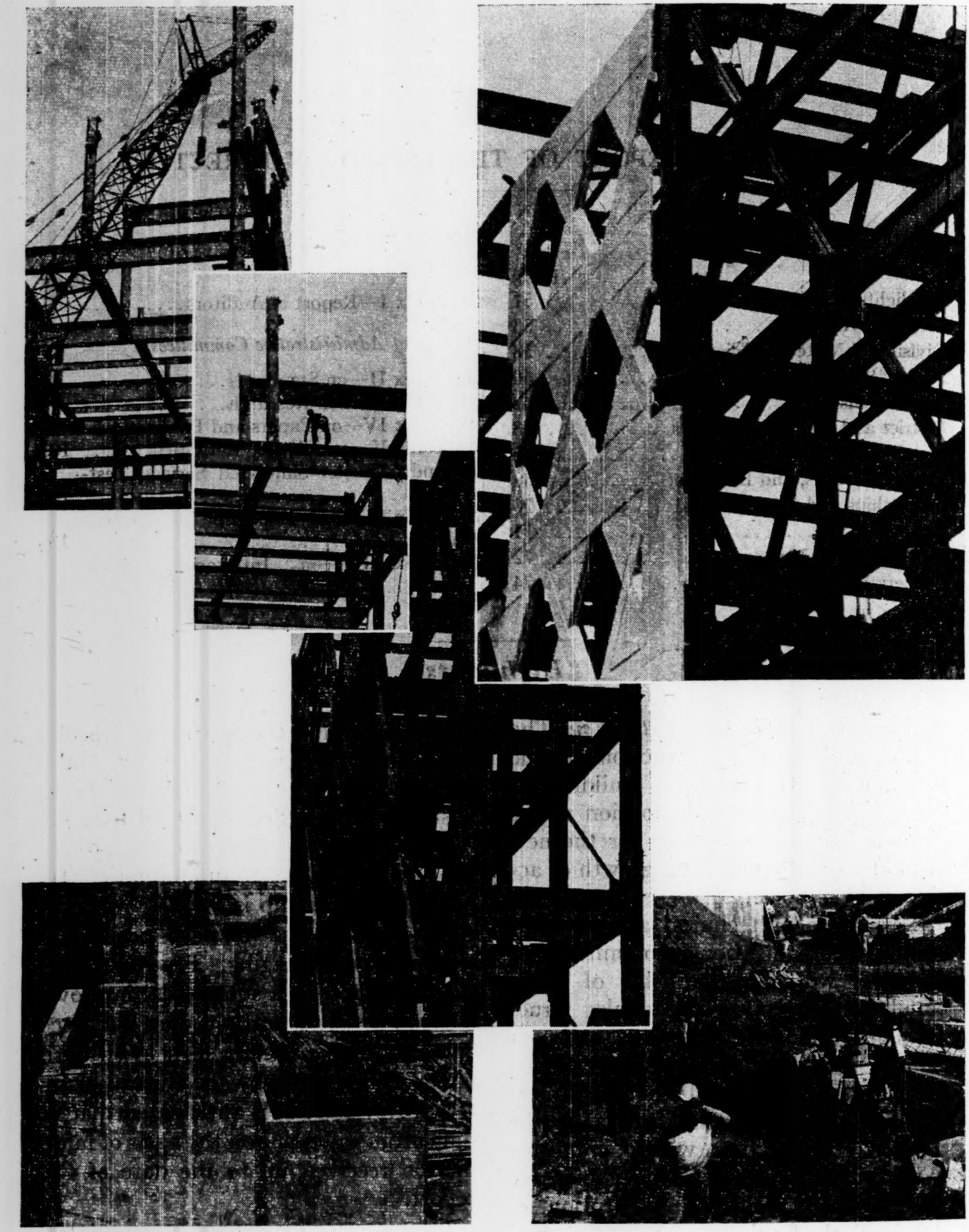
New areas of technical activities included the organization of committees for technical work in the fields of space simulation; pyrolytic materials; structural graphite; peat, peat moss, and peat humus products; and asbestos fibers. Plans were completed for an International Conference on Materials to be held in conjunction with Committee Week in 1964 and for a joint conference with ASME and The Institution of Mechanical Engineers on creep and fracture.

In district activities, there was continued growth with the approval for the formation of a district in Hawaii and the separation of the Southwest District into three districts, namely, the Oklahoma District, the Northern Texas District, and the Southwest District.

In membership, the Society's growth has been steady if undramatic. Its present membership is 11,313.

A review of the financial position is extremely encouraging. The past year closed again with a substantial improvement in actual operations over budgeted operations. The Society's tax position is under examination by the Bureau of Internal Revenue. It is anticipated that no difficulties will ensue on the basis of information received up to the date of this report.

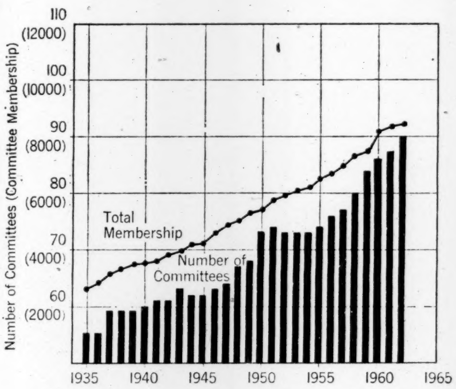
One noteworthy item of broad interest was a decision made by the Board at its meeting in January, 1963, in connection with the metric system. In essence, the Board's resolution calls for the Society to



Step-by-Step Growth of New Headquarters Building assume a position of leadership working with other organizations toward the general national and international development and use of a common system of measurement. As a step in this direction, the Society has decided to provide metric equivalents for other units of measurement in its publications. This decision was widely publicized and many other technical societies have expressed interest and in some cases offered cooperation.

The Board will continue to direct the Society's activities toward maintaining its position of leadership and continued service to the membership, to the general welfare of the nation, and to the industrial economy.

Technical Activities



Growth in Number of Committees and in Official Committee Membership

No attempt is made, under this one heading, to cover completely the technical activities of the Society. Reference should accordingly be made to the sections on meetings, on publications, the discussion on the Division of Materials Sciences, and to the reports of the administrative committees appended to this report, as for example, the Administrative Committee on Research,[1] the Administrative Committee on Simulated Service Testing,[2] and in particular the report of the Administrative Committee on Standards.[3] This latter report discusses in some detail some of the considerations entering into the organization of new committees and ties in quite definitely with a number of the items covered in this report. It also indicates the extent of the standardization activities. It includes, for example, a record of the actions taken at the 1962 Annual Meeting as well as the record of actions by the Administrative Committee on Standards on recommendations from the technical committees during the year. This record shows that the actions at the Annual Meeting resulted in 110 new standards and tentatives, making a total of 3071. During the year, the Administrative Committee on Standards approved 72 new tentatives.

Nor is there any attempt to cover the work of the individual technical committees; for these, references should be made to their annual reports—for the results of investigations sponsored by them and for a report on technical programs that may have been held under their auspices. Some of this has been reported currently during the year in *Materials Research & Standards*. The present discussion, rather, is confined largely to those matters that required action on the part of the Board. Mention should be made, however, of the Conference of Technical Committee Officers held in November 1962, the second of such conferences to be held. These conferences serve a very useful purpose in providing a means for exchange of experiences among the technical committee officers and provide for discussion of various topics concerning the conduct of committee operations that are of particular concern.

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[1] See p. 96.
[2] See p. 105.
[3] See p. 88.

Standardization Procedure:

By action of the last Annual Meeting, the By-laws of the Society were revised to provide for a procedure for the adoption of standards between Annual Meetings, which would be available to committees in addition to the established Annual Meeting procedure. While these changes in the By-laws set forth the basic provisions, namely, that advance announcement should be made to the entire membership concerning any proposed actions with respect to adoption as standard, and that an opportunity to vote on the proposals be provided, certain of the details for implementing the procedure still remained to be developed. This was assigned to a special *ad hoc* committee under the chairmanship of H. C. Miller, together with the review of the proposed revised Regulations Governing ASTM Technical Committees resulting from the recommendations from the Long-Range Planning Committee. Because of the efforts of this *ad hoc* committee, it is expected that the complete interim procedure for adoption of standards will be available during the coming year. Basically this would provide for action on the part of the Administrative Committee on Standards at three-month intervals during the year.

New Activities:

At no time have there been so many new committees authorized. These run the gamut of committees on peat, peat moss, and peat humus products to a committee on space simulation. The various subjects covered, together with the appropriate scopes assigned, are set forth below.

Temperature Measurement (*Committee E-20*).—On the recommendation of Committee E-1 on Methods of Testing, the activities of two subcommittees, one on thermometers and the other on thermocouples, have formed the nucleus of a new technical committee on temperature measurement in recognition of the importance of this fast growing field. The scope approved for this committee follows:

Scope: To promote the knowledge and advancement of the science of temperature measurement by:

(*a*) Aiding and advising the technical committees of the Society in the preparation of standard methods and in the development of apparatus;

(*b*) Sponsoring technical meetings and symposia independently or in cooperation with other organizations;

(*c*) Promoting research and development on applicable methods and associated materials;

(*d*) Formulating specifications and methods of test for temperature measuring apparatus; and nomenclature;

(*e*) Assembling a consolidated source book covering all aspects relating to accuracy, application, and usefulness of thermometric methods; and

(*f*) Coordinating the committee activities with those of other relevant ASTM committees and other organizations.

Manufactured Carbon and Graphite Products (*C-5*).—A new committee on manufactured carbon and graphite products was authorized to extend the work of the Society in this general field. This work had started in Committee C-21 on Ceramic Whitewares and Related Products, and several subcommittees are already functioning in the area of carbon graphite seals, nuclear grade graphite, and on pipe. The scope assigned to this committee is as follows:

Scope: The promotion of knowledge, stimulation of research, and the development of methods of test, specifications, and nomenclature for manufactured carbon and graphite products.

These activities will be coordinated with those of other related committees of ASTM and other organizations (specifically ASTM Committees C-6, C-8, D-1, D-2, and D-24).

Activated (Active) Carbon (Committee D-28).—In view of the need for methods for evaluating activated carbon, a new committee has been established to deal with methods of test for this material. The scope assigned is as follows:

Scope: The development of definitions of terms and nomenclature, methods of test for activated (active) carbon, and the promotion of research in this field. These activities will be coordinated with those of other ASTM committees and other organizations.

Space Simulation (Committee E-21).—The request for the establishment of a new committee on space simulation was an outgrowth of the Third Annual Symposium on Space Environmental Simulation held in June 1962. This subject is of interest to a number of organizations and the interest of these organizations was explored. In the light of the explorations, the Board authorized the establishment of a committee with the following scope:

Scope: The development of definitions of terms and nomenclature, methods of test, and specifications for simulated space environment testing of vehicles, components, and materials intended for space applications and missions, and the promotion of knowledge in this field.

The committee will also function as a coordinating agency maintaining liaison with other organizations and ASTM committees working in this field.

Pyrolytic Materials (Committee C-6).—The need for a committee to deal with pyrolytic graphite had been expressed, and after exploration the subject was broadened so that the Board has authorized formation of a new committee on pyrolytic materials with the following scope:

Scope: The promotion of knowledge, stimulation of research, and the development of methods of test, specifications, and nomenclature for pyrolytic materials.

These activities will be coordinated with those of other related committees of ASTM and other organizations.

Asbestos Fibers.—While various committees of the Society have dealt with asbestos products as, for example, Committee D-13 on Textile Materials, Committee C-16 on Thermal Insulating Materials, and Committee C-17 on Asbestos-Cement Products, there had been no concerted work on asbestos fibers as such. The Board has authorized a conference to establish the interest in a new committee to include other nonmetallic inorganic materials in addition to asbestos fibers.

Peat, Peat Moss, and Peat Humus Products.—In view of the very strong representation from the industry expressing a need for suitable methods for evaluating peat and peat products, which was supported by the consumers, the Board has authorized the establishment of a new committee in this field with the following assigned tentative scope:

Scope: The development of standards relating to peat, peat moss, and peat humus products.

Revised Committee Scopes, Titles, and Jurisdiction:

Technical committees frequently have occasion to review their scopes in view of demands that their activities be broadened, apart from the question of the need for clarification at times so as to avoid conflict of jurisdiction. The following actions have been taken:

Committee D-8.—Change the title and scope to read as follows:

> *Title:* Committee D-8 on Bituminous and Other Organic Materials for Roofing, Waterproofing, and Related Building or Industrial Uses.
>
> *Scope:* The formulation of specifications, methods of test, and definitions of terms pertaining to bituminous and other organic materials for roofing, waterproofing, and related building or industrial uses.

This change was made to enable the committee to take in hand work on specifications for synthetic polymer roofing solutions. The work of this committee will be coordinated with that of Committee D-1 on Paint, Varnish, Lacquer and Related Products and Committee D-11 on Rubber and Rubber-Like Materials.

Committee C-14 on Glass and Glass Products.—Change the scope to read as follows:

> *Scope:* The development of nomenclature, methods of analysis and testing, and specifications for glass and glass products.
>
> Glassware for laboratory testing is covered by other technical committees.

The change consists in the extension of scope to include specifications, which was a departure for the activities of this committee. It was prompted largely by a request from the chemical industry for specifications for glass pipe.

Committee C-20 on Acoustical Materials.—Change the scope to read as follows:

> *Scope:* The study of the properties of materials used to absorb airborne sound, and of the acoustical properties of building materials and structures; the stimulation of research; and the development of methods of test, specifications, and definitions of terms relating to acoustical materials and acoustical properties of materials and structures.
>
> These activities will be coordinated with those of other relevant committees of ASTM, Committee E-6 in particular.

The significant change here is the inclusion of reference to acoustical properties of structures.

Committee D-9 on Electrical Insulating Materials.—Change the scope to read as follows:

> *Scope:* The development of test methods, definitions, and specifications, and the promotion of research relating to electrical insulating materials. Work in overlapping areas will be coordinated with other committees.

This is a simplification of the scope as previously given.

Committee D-16 on Industrial Aromatic Hydrocarbons.—Change the scope to read as follows:

> *Scope:* Formulation of nomenclature and definitions, specifications, and methods of test for industrial aromatic hydrocarbons and heterocyclic chemicals generically classed as coal chemicals and related materials, singly, or as mixtures, and their derivatives.

This modification of scope represents an expansion so as to include products of a nonaromatic nature but produced from aromatic hydrocarbons.

Considerations entering into the review of this statement of scope indicate a need for a general review of the scope of technical committees, since there is considerable variance in the extent to which some of the committees include the considerations of raw materials entering into the product covered by the committee and also in extension of activities to include end products.

Committee E-2 on Emission Spectroscopy.—Change the scope to read as follows:

> *Scope:* (*a*) Formulation, review, and coordination of methods for spectrochemical

analysis by emission and atomic absorption spectroscopy of metallic and nonmetallic materials. Methods include sampling and test procedures used in determining composition.

(*b*) Preparation of general recommended practices relating to emission and atomic absorption spectroscopy.

(*c*) Establishment of nomenclature and definitions relating to emission and atomic absorption spectroscopy.

(*d*) Cooperating with duly constituted agencies concerned with preparation and distribution of standard samples.

(*e*) Advancement of the field of emission and atomic absorption spectroscopy by promoting the collection and exchange of information through (1) surveys and recommendations on related materials and equipment, (2) cooperative test programs, (3) collection and publication of suggested methods and practices, (4) study of unsolved problems, (5) meetings, and (6) symposia.

These activities will be coordinated with those of other relevant ASTM committees and other organizations.

Committee E-11 on Quality Control of Materials.—Change the scope to read as follows:

Scope: To promote the knowledge and use of statistical methods and quality control methods in the specification, control, and evaluation of the characteristics of materials and products. Its primary purpose is to advise ASTM committees in these areas but it is also intended to provide information for more general application.

The committee is authorized to sponsor papers and discussions, to prepare reports, manuals, and recommended practices, and to aid and advise the committees of the Society on the application of statistical and quality control methods in: (*a*) the collection, analysis, interpretation, and presentation of data; (*b*) the development and evaluation of test methods; (*c*) establishing specified limits in specifications; (*d*) preparing acceptance sampling plans to be used for determining conformity to specifications; and (*e*) setting up procedures for control of quality during manufacture.

Adjustment of Jurisdiction of Committees A-1 on Steel and A-10 on Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys:

A special committee was set up by the Board under the chairmanship of Vice President I. V. Williams to review questions resulting from the overlap in scope of these two committees. This special committee included representation from both of the technical committees in question. Agreement was reached to recommend to the Board the establishment of a new committee on tubular products. Work in this field would be transferred to the new committee from the present committees. The Board has asked Committees A-1 and A-10 to hold a conference for organizing a new committee on tubular and related products used in piping structures, with Committees A-3 and A-7, and the Joint Committee on Effect of Temperature participating in the conference.

Special Committee on Numerical Data:

Organization of the committee, which was partially effected in May 1962, has been completed during the year with the following officers:

Chairman: William L. Fink,
Vice Chairman: Marshall V. Otis,
Secretary: Frank Y. Speight.

The scope of the committee is as follows:

Scope: (*a*) Encourage accumulation, evaluation, and dissemination of data on pure substances and commercially important materials.

(*b*) Improve communications among ASTM groups and others working in this general area.

(*c*) Encourage use of standard methods for presentation of data.

The committee is reviewing ASTM continuing numerical data projects and is assisting certain committees to expand their programs. Special attention is being given to the problems of financial support of data projects to supplement volunteer efforts in searching the literature and coding data for publication in data collections.

The committee participated in arrangements for a Symposium on Storage and Retrieval of Analytical Data, sponsored jointly by ASTM and the Society for Applied Spectroscopy, held at the Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy. Plans are going forward for a Symposium on Storage and Retrieval of Mechanical Property Data on Materials for some time in 1964.

The committee has accepted the responsibility for "monitoring the area of engineering data documentation and handling and to recommend appropriate actions to the Engineers Joint Council," as requested by the EJC Information Systems Committee.

Improved Relations with American Petroleum Institute:

A very significant development has taken place during the year looking toward the coordination of the work of ASTM Committee D-2 on Petroleum Products and Lubricants and the API Petroleum Fluid Measurement Committee (PFMC). This collaborating arrangement is intended to (1) eliminate duplication of effort in the development and promulgation of standards in the field of petroleum measuring, sampling, and testing and (2) consolidate into single methods those existing methods that are duplicated by both API Standards and ASTM Methods.

It is the expectation that single consolidated standards and methods will be ready by the end of this year. These standards will be referred to Sectional Committee Z-11 looking toward their approval as American Standard.

Division of Materials Sciences

Election of Officers:

Richard T. Kropf has been elected chairman of the Division for a term of two years succeeding K. B. Woods who had been chairman of the Division from the time of its organization in 1960. John D. Sullivan was elected vice-chairman and the following members of the Executive Committee were elected:

A. Allan Bates
A. G. H. Dietz
W. L. Fink
J. J. Harwood
F. L. LaQue
B. J. Lazan
R. E. Peterson
I. V. Williams
K. B. Woods
L. L. Wyman

Division Membership:

Total membership of the Division, including the Executive Committee and Council, as of April 9, 1963, was 334.

Publications:

During the year, the Society published, on behalf of the Division, two Special Technical Publications in the Materials Sciences Series—Symposium on Stress-Strain-Time-Temperature Relationships in Materials, *STP 325*, and Symposium on Properties of Surfaces, *STP 340*. The publication of the Symposium on Dynamic Behavior of Materials, comprising sixteen papers presented under joint auspices of the University of New Mexico and the ASTM Rocky Mountain District in September 1962, has also been endorsed by the Division as one of the Materials Sciences Series.

Symposium Plans:

The Division is sponsoring a Symposium on Recent Advances in Adhesion Science at the Annual Meeting of the Society in June 1963.

The Division is cooperating in the development of the program for the International Conference on Materials to be held in February 1964 in Philadelphia and is developing a two-session

Symposium on Irreversible Effects Produced by High-Pressure Treatment of Materials.

The topic selected for a Division-sponsored symposium at the Annual Meeting, 1964, is Internal Friction and Damping.

Materials Sciences Luncheon:

For the second successive year, the Division has sponsored a Materials Sciences Luncheon at the Society's Committee Week Meetings. A special feature of Committee Week in February in Montreal was the Luncheon Address by Dr. John Convey, Director, Mines Branch, Canadian Department of Mines and Surveys, on the topic "Materials Research in Canada."

Materials Education:

The Division continues to concern itself with materials education and is counseled by a committee of three consisting of M. J. Sinnott, chairman, T. J. Dolan, and J. J. Harwood. The committee will advise the Executive Committee on policies with regard to Division activities in the area of materials education. In a survey of members of the Division, a large majority of the members expressed the view that the Division should be concerned with materials education in universities, either by influencing curricula or by sponsoring special programs.

Publication Policies:

Based on a recommendation from the Division's Committee on Publication Policies, action has been taken toward the establishment of a special column in *Materials Research & Standards* devoted to "Materials Sciences Notes." The objective of this proposed new department of the magazine is to provide for relatively quick announcement of research findings in the area of materials sciences.

The Materials Sciences Series of Special Technical Publications (referred to above) has been well received and is to be continued as a regular activity of the Division.

Acta Metallurgica:

Action has been taken to become a full joint sponsor of *Acta Metallurgica* with the American Society for Metals and the American Institute of Mining, Metallurgical and Petroleum Engineers. The Society will be represented on the governing body of *Acta Metallurgica* and will have a voice in publication policies. Members of the Society will continue to have the privilege of subscribing to *Acta Metallurgica* at a reduced rate as previously when the Society was affiliated as a cooperating society. The scope of *Acta Metallurgica,* which covers both metallic and nonmetallic solids, appeared in the report of the Division in 1962.[4]

Cooperation with ASTM Districts:

Most of the ASTM Districts are represented on the DMS Council and the Districts are encouraged to sponsor regional interdisciplinary symposia on materials sciences in cooperation with universities.

International Conference on Permafrost:

On behalf of the Division, the Society is cooperating with a number of other organizations in the sponsorship of an International Conference on Permafrost to be held at Purdue University on Nov. 11–15, 1963. K. B. Woods, Past Chairman of the Division, is general chairman of the conference, the purpose of which is to foster an exchange of scientific and engineering significance with primary emphasis on permafrost; to correlate and evaluate current research information and programs; to identify areas that need research; and to stimulate accelerated research efforts.

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[4] 1962 Annual Report of the Board of Directors, *Proceedings*, Am. Soc. Testing Mats., Vol. 62, p. 63 (1962).

National and Other Meetings

1962 Annual Meeting:

The Sixty-fifth Annual Meeting, held in New York, N. Y., the week of June 24–29, 1962, had an attendance of 3291, with 256 ladies also registered as attending. Over 1000 meetings of technical committees were held in addition to 39 technical sessions, the President's Luncheon, the Awards Luncheon, and the Aerospace Materials Industry Luncheon. The Division of Materials Sciences again sponsored a special symposium—on Stress-Strain-Time-Temperature Relationships in Materials—held Wednesday morning and afternoon.

The committee reports presented and many of the technical papers were published in the 1962 *Proceedings*. Many of the papers, particularly those in symposia, were published as Special Technical Publications (STP's). Several of the papers presented at the meeting appeared in *Materials Research & Standards*. A review of the meeting appeared in the July, 1962, issue of this journal.

District Support.—The Society is indebted to the New York District for acting as hosts. The ladies appreciated a most interesting entertainment program prepared for them, and the members and their wives found the Social Hour on Wednesday evening a welcome break in a week of most intensive meetings.

Materials Testing Exhibit and Photographic Exhibit.—The meeting also featured the very interesting exhibit of Materials Testing Equipment and the fine Photographic Exhibit. This represents the fifteenth apparatus exhibit the Society has sponsored in connection with annual meetings.

1963 Committee Week:

The first National Meeting of the Society outside of the United States was held in Montreal, Canada, February 4 to 8, at the Queen Elizabeth Hotel; some 200 committee meetings were held throughout the week.

This first Canadian Meeting of ASTM had the distinction of featuring the First ASTM Lecture on Outstanding Research. This lecture was instituted by ASTM in 1962 for the purpose of having scientists and industrial research men who have personally made outstanding contributions to scientific knowledge discuss the major research in their fields. This first lecture was presented by Cyril Stanley Smith, Institute Professor, Massachusetts Institute of Technology. His topic was "Some Outstanding Historical Metallurgical Research."

The meeting also featured a Luncheon sponsored by the Division of Materials Sciences where Dr. John Convey of the Canadian Department of Mines and Technical Surveys was the guest speaker. In this, too, the First Canadian Meeting has the distinction of having had the largest attendance at any luncheon sponsored by ASTM—475. The week also included a reception on Wednesday morning for Canadian members.

In all, the Society is much indebted to the General Committee on arrangements, composed of Canadian members from the vicinity of Montreal, for their efforts in being hosts to the Society's first Canadian meeting.

Committee Officers Conference:

Over 100 officers of the Society's technical committees attended the two-day conference held in Philadelphia, Nov. 19 and 20, 1962. This is the second conference of committee officers held, now an established part of ASTM activity. Future conferences will be held biennially in even-numbered years corresponding with the election of new committee officers.

The conference reviewed a number of critical topics that will affect the future operations of the Society and its technical activities. At the opening session both national and international standardization were discussed, including ISO and Pan American standards. This was followed by work-shop sessions for secretaries and chairmen, at which details of committee operations were considered. The final session took up a number of questions of broad interest to the Society.

Fourth Pacific Area National Meeting:

Another most successful Pacific Area Meeting was held in Los Angeles the week of Sept. 30 to Oct. 5, 1962, with an attendance of 1356 together with some 60 ladies. The technical program consisted of 49 technical sessions, including the extremely well-attended Symposium on Properties of Surfaces sponsored by the Division of Materials Sciences. The meeting also featured four luncheons: The Aerospace Materials Industry Luncheon, held on Monday, Oct. 1, was addressed by Frank W. Fink, Vice President, Sales and Engineering, Ryan Aeronautical Co. The Wood Industry Luncheon was held on Tuesday, Oct. 2; Alan T. Smith, Manager, Soft Wood Lumber Products, Weyerhaeuser Co., was the guest speaker. Wednesday, Oct. 3, featured the President's National Luncheon which was addressed by Robert L. Minkler, Chairman of the Board of Trustees, California Institute of Technology. The Soils Engineering Luncheon was held on Thursday, Oct. 4, and had as its speaker F. P. Shepard, Professor, Scripps Institution of Oceanography.

In addition to the outstanding technical sessions and luncheons, the Fourth Pacific Area Meetings included over 100 meetings of technical committees and the Third West Coast Materials Testing Exhibit and Photographic Exhibit.

As usual, this Pacific Area meeting proved to be outstanding in every way, for which the Society is greatly indebted to its members on the West Coast and the most efficient and hard working General Committee on Arrangements.

Other National Meetings:

In addition to the above regular National meetings the following National meetings were held:

Joint ASTM-Navy Symposium on Standards for Filament-Wound Reinforced Plastics held on June 6–7, 1962, at the U. S. Naval Ordnance Laboratory, Silver Springs, Md. It was sponsored by the U. S. Navy and Subcommittee XVIII on Reinforced Plastics of ASTM Committee D-20 on Plastics.

Symposium on Dynamic Behavior of Materials held on Sept. 27–28, 1962 at the University of New Mexico, Albuquerque, N. Mex., jointly sponsored by the University of New Mexico and the ASTM Rocky Mountain District.

General Meeting on the Practice of Gas Chromatography sponsored by ASTM Committee E-19 on Gas Chromatography in cooperation with the Michigan State University Program for Continuing Education, on Sept. 26 to 28, 1962, at the Kellogg Center for Continuing Education, at Michigan State University.

The objective of this three-day meeting was to bring together experts on the technique of gas chromatography to discuss the utility and practical value of this technology.

Tenth Annual Meeting—ASTM Committee E-14 on Mass Spectrometry:

This meeting was held on June 3–8, 1962, in New Orleans, La., with an attendance of 400, and consisted of nine technical sessions covering Hydrocarbon Studies, Analytical Techniques, Instrumentation, Negative Ion Symposium, Collision Processes, and Solids Techniques. This regular meeting of ASTM Committee E-14, the only major meeting in this field in the country, covers all phases of mass spectrometry from the theoretical to the practical.

International Conference on Materials—1964 Committee Week:

The International Conference on Materials will be held Monday through Thursday, Feb. 3 to 6, 1964, during the 1964 ASTM Committee Week in Philadelphia. Four symposia and a closing session have been developed.

Symposium on Application of Advanced and Nuclear Physics to Testing Materials is under joint auspices of ASTM and the International Union of Testing and Research Laboratories for Materials and Structures (RILEM). Six papers are being sponsored by RILEM and six by ASTM.

Symposium on Impact of Physics on the Analysis and Testing of Materials will include six papers.

Symposium on Effect of High Pressure on the Properties of Materials (presented by the Division of Materials Sciences) will include seven papers.

Symposium on Modern Physics and New Materials will include six papers.

The closing session on new fields of materials interest will include papers covering areas not yet under active development by ASTM.

Dedication of New ASTM Headquarters.—Wednesday, Feb. 5, is being set aside for the dedication of the new ASTM Headquarters and an address by a prominent individual in the field of science. The Wednesday evening Society Dinner will include a talk by Isaac Asimov, Boston University School of Medicine.

Future Meetings:

Annual Meetings of the Society have been scheduled as follows:

June 21–26, 1964—Chicago, Ill.
June 13–18, 1965—Purdue University, Lafayette, Ind.
June 26–July 1, 1966—Atlantic City, N. J.
June 25–30, 1967—Boston, Mass.
June 16–21, 1968—San Francisco, Calif.
June 22–27, 1969—Atlantic City, N. J.

Committee Week Meetings are as follows:

Feb. 3–7, 1964—Philadelphia, Pa., with International Conference on Materials
Feb. 8–12, 1965—Cleveland, Ohio
Jan. 30–Feb. 4, 1966—Washington, D. C.
Feb. 5–10, 1967—Detroit, Mich.
Jan. 28–Feb. 2, 1968—Atlantic City, N. J.

Pacific Area National Meetings are scheduled as follows:

Oct. 31–Nov. 5, 1965—Seattle, Wash.
June 16–21, 1968—San Francisco, Calif. (Annual Meeting)

Conference of Technical Committee Officers:

Nov. 23–24, 1964—Philadelphia, Pa.
Every even-numbered year.

Publications

Referring to the appended report of the Administrative Committee on Papers and Publications,[1] particular attention is called to the number of special publications that the Society is issuing. The report records the regular publications and also the large number of the so-called Special Technical Publications (STP's) that have resulted from the various meetings of the Society, but apart from these more or less regular publications, the technical committees are sponsoring a number of such special publications as the indexes to data in the infrared and ultraviolet spectrographic field sponsored by Committee E-13 on Absorption Spectroscopy and the Coden book of technical journals also sponsored by that committee, manuals on specific subjects, and bibliographies in specific fields compiled under the auspices of the various technical committees. In a sense they comprise a separate series of Society publications and may need to be recognized as such.

Book of Standards:

The largest single publication effort of the Society is the publication of its standards. A major realignment is in prospect beginning with the 1964 edition, as mentioned in the report a year ago. The complete standards will be published annually. The final plans call for issuing the book in 32 parts corresponding roughly to the compilations that have been issued in various areas. Some few compilations covering broad fields of interest and not corresponding directly to some part of the Book of Standards will still be issued. This new method of publishing the Book of Standards will entail considerable readjustment in the method of distribution and merchandizing; this is receiving the attention of the Finance committee and is referred to elsewhere in this report.

District and Related Activities

During the year, four new districts became active—Mexico, North Texas, Oklahoma, and Hawaii. A fifth new district is under consideration for the Mississippi Delta area. The total number of active districts is now 24.

A significant trend toward technical symposia (from one to two days in length) sponsored by the districts was evident this year. Five such symposia were held, compared to two the previous year. Those held during the year were sponsored by the New England District, the Rocky Mountain District, Central Plains District, Chicago District, and the Northwest District.

Each district, with the exception of two, held at least one meeting during the year. A table of the meetings is attached. Complete news accounts of the meetings were reported throughout the year in *Materials Research & Standards*. President Webber spoke before seven districts, while Vice-President Williams spoke at six. Executive Secretary Marshall attended 13 meetings. In all, 32 meetings were held of which 13 were sponsored jointly with other organizations.

Another trend is toward the formation of District Speakers' Bureaus following the example set by the Chicago District. The Middle Atlantic District will create its bureau in the fall of 1963. Two other districts have started work.

The Student Award Program continues to be strong as a district activity, with 15 districts active and three in the process of planning their programs. A total of 629 awards were sponsored by the districts in addition to 200 sponsored by the Board of Directors. Awards were presented at 150 schools throughout Canada, United States, and Mexico.

The New York District was host at the 1962 Annual Meeting. The Southern California, Northern California, and Northwest Districts joined to make arrangements for the Fourth Pacific Area National Meeting in Los Angeles.

Districts continue to be active in such local technical affairs as Science Fairs, Technical Councils, and celebrations of Engineers Week. The Mexican District translated into Spanish the new ASTM membership brochure for use by the Society in Spanish speaking countries. Also the New England District has undertaken a membership campaign on behalf of the Society. The Northern California District cooperated with the Department of State in hosting the Asian Productivity Group of nine standards experts from eight Far Eastern countries at a one-day orientation session to familiarize the visitors with ASTM.

The ACDA continues its efforts to strengthen the Districts. A survey and open forum at the 1963 Annual Meeting is one such effort (see ACDA report[5]).

District Meetings:

District	*Date*	*Place*
Central New York Materials for Supersonic Transport Aircraft	Oct. 19, 1962	Syracuse, N. Y.
Central New York Clothing	March 29, 1963	Albany, N. Y.
Central Plains (jointly with AIA, CSI, ASCE, ASME, Kansas City University) ASTM and Construction Industry	April 24, 1963	Kansas City, Mo.
Chicago (jointly with BCE, AIA, CSI, WSE and Producers Council) Building Materials	May 2, 1963	Chicago, Ill.
Cleveland Satellite Communications Systems	March 21, 1963	Cleveland, Ohio
Detroit (jointly with ESD) Space Age Materials	Nov. 14, 1962	Detroit, Mich.
Detroit Metals in Sea Cables	April 24, 1963	Detroit, Mich.
Hawaii Inaugural Meeting	April 5, 1963	Honolulu, Hawaii
Hawaii Business Meeting	June 7, 1963	Honolulu, Hawaii
Mexico Inaugural Meeting	Oct. 22, 1962	Mexico City, Mexico
Mexico International Standards for Plastics	March 4, 1963	Mexico City, Mexico
Middle Atlantic (jointly with ASM) High-Temperature Corrosion	April 15, 1963	Baltimore, Md.
Mississippi Valley (jointly with Engineers Club) Metals in Sea Cables	Oct. 18, 1962	St. Louis, Mo.
New England (jointly with ASME) Properties of Plastics	Oct. 18, 1962	Cambridge, Mass.
New England Material Sciences	May 10, 1963	Storrs, Conn.
New York International Standards for Plastics	Dec. 12, 1962	New York, N. Y.
New York The Automotive Challenge to Chemistry	April 30, 1963	New York, N. Y.
Northern California International Standards for Plastics	March 7, 1963	San Francisco, Calif.
Northern California Atomic Power	April 3, 1963	San Francisco, Calif.
Northern Plains Satellite Communications Systems	Oct. 17, 1962	Minneapolis, Minn.
Northern Plains (jointly with NAPE) The New North Star	June 10, 1963	Minneapolis, Minn.
North Texas Inaugural Meeting	March 19, 1963	Dallas, Texas
Northwest (jointly with ACI and Oregon State University) Concrete	March 21–22, 1963	Corvallis, Ore.

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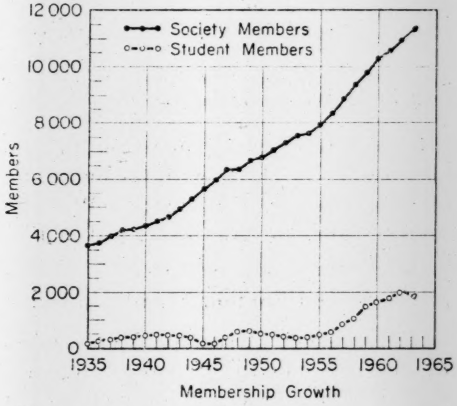
[5] See p. 103.

District Meetings (*Continued:*)

District	*Date*	*Place*
Ohio Valley (jointly with Committees F-2, D-10) Packaging	Oct. 2, 3, 4, 1962	Columbus, Ohio
Oklahoma Inaugural Meeting	April 26, 1963	Tulsa, Okla.
Philadelphia Plastics in Daily Use	Oct. 29, 1962	Princeton, N. J.
Philadelphia Company Standards	April 23, 1963	Newark, Del.
Pittsburgh (jointly with AWS, ARS, ASM) Welding for Space Applications	Nov. 29, 1962	Pittsburgh, Pa.
Pittsburgh Satellite Communications Systems	April 23, 1963	Pittsburgh, Pa.
Rocky Mountain (jointly with University of New Mexico) Dynamic Testing	Sept. 27–28, 1962	Albuquerque, N. Mex.
Southern California (jointly with SPE, SPI) International Standards for Plastics	March 5, 1963	Los Angeles, Calif.
Southwest (jointly with SPE) Polymers	March 1, 1963	Houston, Tex.

Membership



Growth in Membership

New members continue to join the Society at more than a thousand per year. In the period May 1, 1962, to May 1, 1963, more applications were accepted than in any previous year—1287—as compared with 1141 in 1961, the previous high year. However, net growth is always substantially less because of losses from death, resignation, and nonpayment of dues. The net gain in new members was 418 in all classes except student, as compared with 390 a year ago. There was a net loss in student membership of 293.

While the total membership moves steadily upward, it continues to be a matter of concern to the Board that the relative growth of the Society in members is not keeping abreast of the growth of the scientific and engineering activities in our economy nor with the growth of our technical committees. Of particular concern is that for the third straight year there has been a drop in the number of company members. This is attributed to the fact that since company member dues were raised in 1959 a number of companies have resigned their company memberships and provided individual memberships for their former company representatives. Another factor contributing to the reduction in company members is that some large companies have combined two or more company memberships into a single sustaining membership.

The rate of membership growth is expected to accelerate as a result of a reinforcement in staff introduced in the spring of 1963.

On May 1, 1963, the membership of the Society totaled 11,313, compared with a total of 10,895 one year ago. Gains and losses in various classes of membership are shown in the following table:

Class of Membership	Hon orary	Perpetuity and Life	Sustaining	Industrial	Institutional	Personal	Associate	Total	Student
Total Membership May 1, 1962	31	14	439	2022	[a]	7926[a]	463	10 895	1989
Add:									
New Members	2		5	107		821	97	1032	816
Reinstatements or Renewals			1	5		39	7	52	
Transfers to:	6	4	1	1		102	197	311	
Total Additions	8	4	7	113		962	301	1395	816
Deduct:									
Deaths	2					58	1	61	1
Resigned			4	77		375	20	476	511
Dropped			1	38		204	89	332	394
Transfers from:		3		4		13	88	108	203
Total Losses	2	3	5	119		650	198	977	1109
Net Change	6	1	2	−6		312	103	418	−293
Total Membership May 1, 1963	37	15	441	2016	954[a]	7284[a]	566	11 313	1696

[a] Institutional class of membership formed late 1962. The total number of this class on May 1, 1963, is 954; these members were formerly included in the personal class of membership.

It is interesting to compare out present membership of 11,313 with the figures for ten and twenty years ago; in 1953 our total membership was 7522, and in 1943 it was 4904.

Associate and Student Memberships:

The class of Associate Membership, which continues to 30 years of age and replaces the former junior membership with its 27-year age limit, continues to grow as noted in the table.

The reduction in student memberships is considered to be of a transitory nature.

Sustaining Members:

Seven new sustaining members offset the five losses in this class. The new sustaining members are:

Aerojet-General Corp.
Atomics International, A Division of North American Aviation, Inc.
Bell Telephone Laboratories, Inc.
Chemical Research and Development Center, FMC Corp.
The Collison Surgical Engineering Co.
Dodge Brothers (Britain) Ltd.
Rohr Corp.

60-Year Members:

Four organizations this year join the list of those which have been members continuously for 60 years:

The Carpenter Steel Co.
Arthur D. Little, Inc.
National Lead Co.
Pennsylvania, Commonwealth of, Department of Highways

50-Year Members:

There are four individuals and nineteen organizations as noted below who this year have been members of the Society continuously for 50 years:

John M. Bierer
John J. Crowe
Ralph E. Roscoe
L. H. Winkler

American Car and Foundry Division, ACF Industries, Inc.

The Atlantic Refining Co.
Combustion Engineering, Inc.
Compressed Gas Assn., Inc.
Giant Portland Cement Co.
Hydro-Electric Power Commission of Ontario
Leeds & Northrup Co.
Missouri School of Mines and Metallurgy
Northern Pacific Railway Co.
The Ohio Brass Co.
Otis Elevator Co.
The Pennsylvania Railroad Co.
University of Queensland
Rutgers, The State University, School of Ceramics
Seattle City Engineer's Dept.
The A. P. Smith Manufacturing Co.
Tennessee Coal and Iron Division, United States Steel Corp.
City of Toronto, Department of Public Works
Union Carbide Metals Co., Division of Union Carbide Corp.

40-Year Members:

There are 64 members who this year have completed 40 years of membership:

S. Frank Cox
W. H. Graves
Gerald J. Horvitz
Maxamillian A. Krimmel
Nolan D. Mitchell
S. M. Phelps
Edgar S. Ross
E. W. Schultheis
John M. Weiss
J. A. Williams
Hubert Woods

City of Baltimore, Bureau of Tests
Bemis Bro. Bag Co.
University of Birmingham Library
The British American Oil Co., Ltd.
The Buffalo Slag Co., Inc.
Coplay Cement Manufacturing Co.
Copper & Brass Research Assn.
The Cotton Silk and Man-Made Fibres Research Assn.
The Electric Storage Battery Co.
Electrolytic Refining and Smelting Company of Australia, Pty., Ltd.
The Ensign-Bickford Co.
The Fulton Sylphon Division, Robertshaw-Fulton Controls Co.
The Gaertner Scientific Corp.
A. P. Green Fire Brick Co.
Greiner Scientific Corp.
H-B Instrument Co.
James Jerome Hill Reference Library
Hollingsworth & Vose Co.
University of Idaho, Civil Engineering Dept.
Imperial Oil, Ltd.
Johannesburg Public Library
Johns-Manville Dutch Brand Products Corp.
Kansas City Water Dept.
Koninklijke/Shell Laboratorium, Amsterdam
Laclede Gas Co.
Laclede Steel Co.
Link-Belt Co.
University of Liverpool
City of Los Angeles, Department of Building and Safety
Medusa Portland Cement Co.
Metal Finishing
University of Michigan, General Library
The New Jersey Zinc Co.
New South Wales, Department of Railways
New York State Department of Public Works, Division of Architecture
Ohio Lime Co.
Oldsmobile Division, General Motors Corp.
Pacific Gas and Electric Co.
Pittsburgh-Des Moines Steel Co.
Precision Scientific Co.
Riverside Cement Co., Division of American Cement Corp.
Rochester Engineering Society
University of Rochester Library
Sangamo Electric Co.
Sargent & Lundy
Seaside Oil Co.
Shell Oil Co., Inc.
South Carolina State Highway Dept.
Standard Railway Equipment, Division of Stanray Corp.
Taylor Forge and Pipe Works
Van Cleve Laboratories, Inc.
Walworth Co.
The Whitehall Cement Manufacturing Co.

Deaths:

The Society has lost the following members through death (figures in parentheses are dates of membership):

Benjamin A. Anderton (1914)
James Aston (1912)
H. Austill (1908)
Lincoln B. Barker (1956)
Terrell Bartlett (1936)
C. W. Bek, Jr. (1951)
Earle G. Benson (1962)
G. B. Boon (1930)
Carl D. Brandt (1937)
J. Ed. Brewer (1924)
Don Brouse (1961)
Allan B. Campbell (1949)
A. C. Chamberlin (1936)
G. H. Clamer (1902)
Robert A. Cummings (1904)
Christian Dantsizen (1946)
Donald Thomas Davidson (1950)

W. E. Day, Jr. (1923)
Edgar Hutton Dix (1919)
V. L. Duval d'Adrian (1951)
Henry F. Eagan (1961)
W. L. Gilmer (1952)
Louis Glaberson (1961)
Howard C. Hardy (1962)
Glenn H. Hicks (1949)
E. W. Hindman (1960)
L. T. Holt (1920)
W. W. Johnstone (1947)
Armand E. Keeley (1945)
Potter W. Kelly (1952)
John N. Kenyon (1932)
D. W. Kessler (1920)
C. S. King (1941)
Maurice G. Kramer (1955)
Walter J. Kunkel (1939)
Thomas C. Law (1920)
Robert H. Leach (1926)
Bernard F. Locraft (1951)
Ernest F. Lundeen (1946)
Herbert McClammy (1948)
Daniel P. Norman (1940)
Angelo F. Nunnari (1952)
Henry B. Oatley (1954)
William H. Paul (1952)
Edwin P. Pitman (1940)
Raymond A. Pulk (1962)
W. E. Robinson (1931)
Edgar S. Ross (1923)
S. O. Schamberger (1956)
Hal W. Shidemantle (1962)
F. Allen Simmons (1961)
Earl B. Smith (1906)
Theodore P. Steinmetz (1952)
Oliver W. Storey (1915)
A. G. Streblow (1952)
John S. Sulecki (1960)
J. Roy Tanner (1931)
H. E. Tucker (1952)
C. Benson Wigton (1913)

In addition to the above, the following representatives of company members of the Society passed away:

C. R. Baker, Pacific Tube Co.
O. Marvin Beach, Frank L. Crobaugh Co.
Donald A. Campbell, Eclipse Fuel Engineering Co.
Myron B. Diggin, Hanson-Van Winkle-Munning Co.
G. V. Kingsley, Bohn Aluminum and Brass Co.
Wilmer D. Langtry, Commercial Testing and Engineering Co.
John J. Manning, Concrete Industry Board
J. P. McKenzie, Copolymer Rubber & Chemical Corp.
L. J. Muzslay, The Chemical Rubber Co.
W. F. Rhodehamel, Seaside Oil Co.
Fred B. Riggan, Texas Steel Co.
C. J. Rogers, Jefferson County, Ala., Engineering Dept.
H. R. Scott, Delaware Alloy Forge Co.
R. G. Seaman, Rubber World
John W. Whittemore, Virginia Polytechnic Inst., Engineering Experiment Station

Honors, Awards, and Lectures

The Board's Committee on Honors, Awards, and Lectures has been active in reviewing the Society and Committee Awards. A new edition of the Manual on Honors, Awards, and Lectures has been prepared, containing information on the procedure for establishing awards; policies regarding honors, awards, and lectures adopted by the Board; and a complete description of the various Society and Committee Awards and Lectures, with rules governing the selection of the recipients of the awards and the lecturers. Brief descriptions of the various awards as well as the personnel of the lecture and award committees are given in the ASTM Year Book.

Recipients of honors and awards for 1963 are indicated below:

Honorary Membership:

Honorary Membership has been bestowed on the following:

Robert Burns
George H. Harnden
Max Hecht
Francis M. Howell

ASTM Award to Executives:

As mentioned in the report last year, an ASTM Award to Executives has been established to honor an executive who, through his outstanding interest and support, has furthered the accomplishments of ASTM. The first award was made during the Fourth Pacific Area National Meeting in Los Angeles in October, 1962, to Dr. John Fairfield Thompson, Honorary Chairman of the Board, and Chairman of the Executive Committee of The International Nickel Co. of Canada. Dr. Alexander Crawford Monteith, Group Vice-President of Westinghouse Electric Corp., has been selected as the second recipient. The award to Dr. Monteith will be made at the Awards Luncheon during the Annual Meeting in June.

ASTM Award of Merit:

The 1963 recipients of the ASTM Award of Merit are as follows:

W. M. Bertholf
Francisco Chairez
R. E. Copeland
D. I. Finch
D. Halloran
H. K. Hammond
W. C. Hansen
S. H. Ingberg
D. E. Krause
M. A. Pinney
C. E. Proudley
L. F. Rader
E. E. Schumacher
R. B. Smith
J. V. Steinle
J. A. Truitt
C. Zischkau

Recognition of Technical Papers:

Charles B. Dudley Medal (research).—M. R. Meyerson, T. R. Young, and W. R. Ney for their paper "The Development of More Stable Gage Blocks."

Last year the committee was delayed in selecting the recipient, so no Dudley Medal Award was made during the Annual Meeting. Instead the 1962 award was made during the Fourth Pacific Area National Meeting in Los Angeles, October, 1962, to Frederick C. Hull for his paper "Effects of Alloying Additions on Hot Cracking of Austenitic Chromium-Nickel Stainless Steel."

Richard L. Templin Award (testing).—Robert Markovich and Frederick W. Schwartzberg for their paper "Testing Techniques and Evaluation of Materials for Use at Liquid Hydrogen Temperatures."

Sam Tour Award (corrosion).—No recipient selected for 1963.

Sanford E. Thompson Award (concrete, sponsored by Committee C-9).—D. L. Bloem, R. D. Gaynor, and J. R. Wilson for their paper "Testing Uniformity of Large Batches of Concrete."

C. A. Hogentogler Award (soils, sponsored by Committee D-18).—R. K. Bernhard for his paper "Biaxial Stress Fields in Noncohesive Soils Subjected to Vibratory Loads."

Recognition of Outstanding Contributions:

Frank E. Richart Award (concrete and concrete aggregates).—Kenneth B. Woods, Head, School of Civil Engineering, and Director, Joint Highway Research Project, Purdue University, Lafayette, Ind.

Walter C. Voss Award (building technology).—Douglas E. Parsons, Consultant, National Bureau of Standards, Washington, D. C.

Max Hecht Award (industrial water, sponsored by Committee D-19).—J. F. J. Thomas, Head of Industrial Water Section, Mines Branch, Department of Mines and Technical Surveys, Ottawa, Canada.

Harold DeWitt Smith Memorial Award (textiles, sponsored by Committee D-13).—Richard T. Kropf, President, Belding Heminway Co., Inc., New York, N. Y.

Adhesives Award (sponsored by Committee D-14).—C. A. Dahlquist and D. H. Kaelble of Minnesota Mining and Manufacturing Co., St. Paul, Minn.

Committee D-12 Award (soaps and detergents).—No recipient selected for 1963.

ASTM Lectures:

The 1963 *Edgar Marburg Lecture* on "Adhesives—Past, Present, and Future," will be presented on Wednesday afternoon, June 26, 1963, at Chalfonte-

Haddon Hall, Atlantic City, N. J., by Richard F. Blomquist, Chemist, U. S. Forest Products Laboratory, Madison, Wis.

The 1963 *H. W. Gillett Memorial Lecture* on "Trends in the Metallurgy of Low-Alloy, High-Yield Strength Steels" will be presented on Tuesday afternoon, June 25, 1963 at Chalfonte-Haddon Hall, Atlantic City, by J. B. Austin, Administrative Vice-President, Research and Technology, United States Steel Corp., Pittsburgh, Pa.

The first *Lecture on Outstanding Research* was presented during 1963 Committee Week in Montreal, Canada, by Cyril Stanley Smith, Institute Professor, Massachusetts Institute of Technology, Cambridge, Mass., on the subject "Some Outstanding Historical Metallurgical Researches."

N. P. Allen, Superintendent, Metallurgy Division, National Physical Laboratory, Teddington, England, will present the second Lecture on Outstanding Research during the International Conference on Creep to be held in New York, August 24–29, 1963. His subject will deal with some aspects of research on mechanical properties of metals and alloys, particularly at high temperatures.

Fellowships and Grants-in-Aid

The committee, consisting of R. T. Kropf, Chairman; R. A. Schatzel; K. B. Woods; and F. F. Van Atta, secretary, held three meetings during the year—September 12, 1962, January 4, and April 29, 1963 in New York.

At the meeting on January 4, the committee examined 34 applications from the 48 schools that were invited to apply for the ASTM $1000 Grants-in-Aid. The five shown below were selected to receive grants for 1963–1964:

University of California, Berkeley, Calif.:

For a project under the direction of D. W. Fuerstenau to support a study of the size distribution of particles resulting from the single fracture of brittle materials.

Iowa State University, Ames, Iowa:

To support a project under the direction of David R. Wilder to study the thermal properties of high-temperature materials, using a plasma torch as the source of thermal energy; specifically, the development of thin opaque coatings (carbon, carbide, or refractory metals) on ceramic refractories.

University of Michigan, Ann Arbor, Mich.:

To support a project under the direction of Frank E. Richart, Jr., to evaluate the effect on capillary behavior of short-time changes in gravitational forces, as would be induced by the vertical sinusoidal motion of a soil mass. The problem has arisen in the missile field relating to the transient and permanent capillary movements of materials when the gravity field is reduced or for cases of zero gravity fields—for example, the movements of fluid in rocket tanks during periods of weightlessness.

The Pennsylvania State University, University Park, Pa.:

To support a project under the direction of K. Vedam to determine photoelastic properties of materials at very high temperatures.

Purdue University, Lafayette, Ind.:

To support a project under the direction of Brage Golding for a fundamental study of the nature of adhesion.

On April 29, 1963, the committee reviewed 17 applications for the $6500 ASTM Doctoral Fellowship from the 18 schools invited to apply. The Pennsylvania State University was selected to receive the fellowship for the 1964–1965 academic year.

Finances

Report for 1962:

The auditor's report covering the fiscal operation of the Society for 1962 appears in Appendix I.[6]

Statement of Operations:

The Society completed its fiscal year of 1962 in excellent financial condition. The margin of income over expenses was $171,209 as compared with a budgeted figure of $50,050. This includes a provision of $20,000 for estimated loss on the 1962 *Proceedings*, which is furnished to members at less than cost.

Receipts.—This was the first year following the change to a full accrual basis of accounting. Receipts from dues, entrance fees, and the distribution of additional parts of the Book of Standards for the special membership price were about as anticipated, with each item running a little over the income estimates. Sales of the *1961 Book of Standards* were $115,000 (18 per cent) above income estimates, and more than offset the amounts that sales of STP's and other publications fell short. The drop in sales of STP's and other publications is due largely to the fact that fewer of these were published during the reporting period.

Disbursements.—Administrative expenses were roughly 2 per cent over the budget, and Society office expenses were about 1 per cent over the budget. Of special interest is the fact that $11,000 for the founding of the ASTM Executive Award was allotted out of operating expenses rather than from surplus.

The direct expense for producing publications was $916,077, or 89.4 per cent of this budget item. However, the cost of sales under the accrual system was $756,763. This results in part from (1) the capitalization of inventory as the Society converted to the accrual method; (2) payments from reserves for the *Book of Standards* and *Proceedings*, and (3) the fact that as some books are paid for, the cost is transferred to inventory as an asset. Cost of sales is then determined from the unit cost of books sold from inventory.

Balance Sheet:

The balance sheet included in the annual report of the auditors as of Nov. 30, 1962, shows assets including the general, building, and special and designated funds in the amount of $3,116,484 as compared with $3,017,336 a year ago.

Building Fund:

The Assets in the Building Fund at the end of the fiscal year were $718,654. In January 1962, the Board authorized the transfer of $103,325 from General Fund surplus to the Building Fund to retire a loan of that amount from the ASTM General Fund. The buildings at 1916–18 Race St. and 1921–27 Cherry St. were demolished to make way for the construction of the new Headquarters Building, and it is the present intention of the Society that the building at 1921–23 Arch St. also will be demolished. Pursuant to a resolution of the Board of Directors, the carrying value of land was adjusted to $330,000, based on an independent appraisal of the properties as unimproved land. This places the book value of the land in proper perspective in relation to the new building.

On Sept. 14, 1962, the Society entered a contract with David M. Hunt Construction Co. for the construction of the new National Headquarters Building at a cost of $2,082,845. Of this amount, $45,500 had been paid or accrued to Nov. 30, 1962.

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[6] See p. 77.

A commitment has been obtained from Girard Trust Corn Exchange Bank for a construction loan of $1,000,000 to be used as necessary for construction costs until covered by contributions to the Building Fund. This is secured by the investments held in the Investment Fund.

In addition, a commitment has been obtained from the Western Savings Fund Society of Philadelphia for a $500,000 first mortgage loan upon completion of the new Headquarters Building. The term of the loan is to be 25 years, with level monthly payments of $3000 including interest at $5\frac{1}{4}$ per cent.

Contributions to the Building Fund as of Nov. 30, 1962, less solicitation costs totaled $193,068. As of April 30, 1963, contributions received had reached $414,195 in cash, and an additional $270,018 in pledges for a total of $684,213.

Investments:

The operation of the Investment Fund continues without substantial change under the guidance of Girard Trust Corn Exchange Bank as Investment Counsel. The income from investments is paid semi-annually to the General, Building, Research and other funds in accordance with their pro rata equity. As of November 30, 1962, the book value at cost of the securities in the fund totaled $1,016,674. The market value of the securities was $1,151,350, and the total income $39,286. Cash, interest, and dividends receivable bring the total value of the Investment Fund on a cost basis to $1,065,625; on a market value basis $1,200,302.

1963 Budget:

The 1963 Budget, adopted in September 1962 and revised in April 1963, estimates total income as $1,775,000, and total expenses as $1,851,800, with an anticipated operating deficit of about $76,800. This includes a special authorization by the Board for $35,000 to cover the first year's cost of installing a data processing system and the initial phases of converting our membership and committee records.

The budget has been prepared conservatively, and an examination of the operations for the first four months indicates that the anticipated deficit may be reduced considerably by the end of the fiscal year.

BUDGET SUMMARY: 1963
(Dec. 1, 1962 to Nov. 30, 1963)

Operating Income		Per Cent
Dues and entrance fees	$407 500	22.9
Publications	1 146 000	64.6
Miscellaneous	221 500	12.5
Total Operating Income	$1 775 000	100.0
Operating Expense		**Per Cent**
Administrative	$217 000	11.8
Publications	508 500	30.7
Society Office:		
Salaries	685 400	37.0
Other	380 000	20.5
Total Operating Expense	$1 851 800	100.0
Anticipated Operating Deficit	$76 800	

Administrative Matters

Staff:

Continuing its study of staff organization, the Board of Directors authorized additions to the staff and a realignment of some responsibilities to provide for the expanding technical activities and work of the Society.

Additions to the staff included Mr. J. H. Bystrom who has been appointed manager of Promotion and Field Operations reporting to the executive secretary; the appointment of William Pepper, Jr., as assistant editor of *Materials Research & Standards*; David C. Hathaway as an editorial assistant in the Standards Department; William A. Whittier as technical assistant; and F. J. Kushner to supervise the new data processing unit.

The following promotion and assignment of new duties were made: R. E. Hess, associate executive secretary, has been named acting manager, Publication Operations; L. C. Gilbert, associate technical secretary, has been designated assistant executive secretary; in addition, J. W. Caum has been appointed to succeed Mr. Hess as technical secretary.

Executive Secretary Emeritus Robert J. Painter has spent full time on the Building Fund Committee. During Mr. Painter's extended visit to Europe with Mrs. Painter in the spring of 1963, Past President N. L. Mochel, who has been acting as a consultant to the executive secretary on governmental affairs, assumed responsibility for continuing Mr. Painter's work in directing the Building Fund activities.

Data Processing:

In line with the tremendous growth of activity in the Society, the Board of Directors approved a plan for mechanization of member records and certain other staff functions. Equipment has been ordered and the initial conversion phase which involves the mechanization of member and committee member records has been started. Later steps will include the transfer of certain accounting and billing functions and the maintenance of publications inventory to this activity. The plan will include the use of IBM cards in combination with Scriptomatic addressing and, when combined with the changes being made in the manner of production of the *Book of ASTM Standards*, will permit the preparation of dues bills mechanically. It is anticipated that progress will be sufficient to permit the bills for 1964 dues to be prepared in this manner.

Headquarters:

Plans for the construction of new headquarters facilities proceeded with increasing tempo during the year. Following approval by the Board in the spring of 1962, the Society entered into a contract with David M. Hunt Construction Co. for the construction of a new headquarters building on the site of the former headquarters at 1916 Race St., in comformity with plans prepared by the architectural firm of Carroll, Grisdale & Van Alen of Philadelphia. The contracts were signed in September, 1962, and work was immediately started on the demolition of the old buildings, preparation of the site, and the beginning of construction of the new building.

The plans for the new headquarters provide for an auditorium seating 350 persons capable of being divided into two smaller rooms plus two small meeting rooms of 35 to 50 capacity each, a board room and two larger conference rooms of 75 to 100 capacity, three working floors having a combined capacity of 21,600 sq ft plus the addition of certain special offices, elevators, and other service facilities. The foundation and structure were planned so that four additional floors could be added as future needs of the Society dictate.

During the winter the pilings were successfully placed and by early spring the steel work was completed and the placing of precast panels begun. It is anticipated that the building will be completed and ready for occupancy by December 1963. Plans are under way for a dedication ceremony to be held during the International Conference on

Materials in conjunction with Committee Week in February 1964.

In the interim, the staff has been occupying temporary headquarters in the Westinghouse Building at 3001 Walnut St., with a very small force occupying the Arch St. property, which will be converted to parking facilities to serve the new building.

Under the chairmanship of Past President F. L. LaQue, a Building Fund Committee is well under way in an intensive program to raise the monies required for the new building and new equipment. By the spring of 1963, fund raising, with a goal of 2.1 million dollars, had reached the three-quarter million mark. Contributions are being sought from members and committee members as well as those segments of industry that are affected by the Society's activities.

Cooperative Activities

American Standards Assn.:

The Board of Directors passed a resolution urging the technical committees of the Society to submit ASTM standards to the ASA for approval as American Standard. As a result, there has been a substantial increase in the number of ASTM standards that have also been named American Standard.

With the growth of the importance of the European common market and the Latin American common market, ASTM has worked more closely with ASA in developing means for and encouraging more active participation in the activities of the International Standards Organization. A listing of detailed ASTM participation in ISO and other international standards work is included in the Annual Report of the Administrative Committee on Standards appended to this report.[7]

Government Contacts:

Two major areas of importance to the Society were opened during the current year. The first of these was the culmination of a long series of contacts with the Government Services Administration and has resulted in the establishment of close working relationships between the technical secretary and representatives of GSA to implement and accelerate the coordination of Federal and Military Specifications with industry standards. Supplementing this work is the continued activity of Mr. William Healy of the ASA staff working under a contract with the Department of Defense to cross index Military Specifications and American and ASTM Standards. This latter activity is reported monthly in ASA's *Magazine of Standards*.

The second area of importance resulted from the appointment of Dr. J. Herbert Hollomon, formerly director of the General Engineering Laboratories of General Electric Co., as Assistant Secretary of Commerce for Science and Technology and the implementation of certain phases of a study made in 1960 by an *ad hoc* committee of the National Academy of Sciences on "The Role of the Department of Commerce in Science and Technology." These changes have resulted in strengthening the National Bureau of Standards and a general transfer of responsibilities for standards activities from the Office of Technical Services to NBS.

A meeting of the ASTM-ASA Advisory Committee to the National Bureau of Standards on Engineering and Related Standards was held on March 22. The organizational changes at the Bureau and the effect of these changes on both ASTM and ASA were discussed.

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[7] See p. 88.

ASTM was represented by President A. C. Webber, Past President N. L. Mochel, and the executive secretary. ASA was represented by ASA President F. H. Roby, ASA Vice President M. N. Clair, and Roger E. Gay, managing director. Among the NBS staff present was ASTM Past President A. Allan Bates who has recently become Chief of the Division of Building Research at NBS.

The executive secretary and the treasurer met in the Office of Technical Services of the Department of Commerce with representatives of ASA and other Government agencies early in April to pursue the problems concerned with making selected ASTM and American Standards available in Spanish and Portuguese for use in the Latin American countries. Responsibility for translations in the area of safety standards has been assigned to the Department of Labor.

The executive secretary and assistant executive secretary participated in a meeting in the office of Dr. J. Herbert Hollomon to discuss the proposed Civilian-Industrial Technology Program with particular emphasis on the construction industry. The basic program proposes a four-part program with two broad purposes: (*a*) to assist the continued and expanded use of technology for the upgrading of industrial practices and (*b*) to encourage the development of an adequate supply of scientific and engineering personnel for the conduct of industrial research. The four-part program envisioned, and for which funds amounting to 1.25 million dollars in the current fiscal year and 7.5 million dollars in the next fiscal year are being requested, includes (1) support of the training of personnel at universities for industrial research and development, (2) stimulating research in industry institutions, (3) developing industry university extension service, and (4) supporting technical information services that meet industry's specific needs for knowledge about technological activities and development.

Engineers Joint Council:

The executive secretary attended the convention of the Pan American Federation of Engineering Societies (UPADI) in San Juan, Puerto Rico, and participated in a round table discussion on standards. Resolutions growing out of the round table discussion were transmitted to the member countries of UPADI and have resulted in increased interest in the activities of the Pan American Standards Commission.

Mr. E. C. Shuman, ASTM representative on the Technical Planning Committee of Engineers Joint Council, has been responsible for the initial work on a pilot study endeavoring to solve the problems of multiplicity of technical meetings. It is anticipated that out of the activities of this committee will come a definitive plan whereby the various scientific and engineering societies can cooperate in a clearinghouse of information on future meetings designed to reduce the conflicts of subject matter, degree of sophistication, time, and place.

International Contacts:

Pan American Standards Commission.—The technical activities of the Pan American Standards Commission (CPNT) have progressed actively in four fields of interest to ASTM, namely, steel, cement, textiles, and leather.

The U. S. National Committee for ISO TC/17, functioning under the chairmanship of Society Vice President C. L. Kent as a subcommittee of Committee A-1, has been assigned responsibility for coordinating United States participation on all international standards activities, including CPNT. The initial drafts by the working groups on steel have been referred to this committee.

The activity in the field of cement has

been assigned to ASTM's Committee C-1 which was represented at meetings in South America by Bruce Foster of the National Bureau of Standards. Textile activities, which have progressed quite rapidly, are being handled under the USA Committee for ISO TC/38 under the chairmanship of D. S. Hamby and with the active participation of Dr. H. F. Schiefer of NBS, chairman of ASTM Committee D-13. Technical work on leather has been referred temporarily to the Joint ALCA-ASTM Committee on Leather.

Other Activities.—Plans for an International Conference on Creep to be held in New York Aug. 26–29, 1963, and with the discussion to be continued in London, England, Sept. 30–Oct. 4, have been completed. This conference is under the joint sponsorship of ASTM, ASME, and The Institution of Mechanical Engineers (Great Britain).

The Society is sponsoring an International Conference on Materials to be held concurrently with Committee Week in February, 1964. Participating in the International Conference will be the International Union of Testing and Research Laboratories for Materials and Construction (RILEM). The theme of the International Conference will be the "Impact of Modern Physics on Materials."

Respectfully submitted on behalf of the Board of Directors,

A. C. WEBBER,
President.

T. A. MARSHALL, JR.,
Executive Secretary.
June, 1963.

APPENDIX I

REPORT OF THE AUDITORS FOR THE FISCAL YEAR NOVEMBER 29, 1961 TO NOVEMBER 30, 1962

Philadelphia, January 10, 1963

AMERICAN SOCIETY FOR TESTING AND MATERIALS

Philadelphia, Pa.

We have examined the accompanying balance sheet of the American Society for Testing and Materials as of November 30, 1962 and the related statements of operations and surplus of the general fund and building fund, and statements of changes in special and designated funds for the period November 29, 1961 to November 30, 1962. Our examination was made in accordance with generally accepted auditing standards, and accordingly included such tests of the accounting records and such other auditing procedures as we considered necessary in the circumstances.

As of November 28, 1961, when the Society changed to the accrual basis of accounting, and as of November 30, 1962, no provision was made for the estimated cost (printing cost only) of supplying the 1961 Book of Standards and the 1962 Book of Standards Supplements, respectively, to members electing to receive such volumes without additional charge. In our opinion, such a provision is necessary in accordance with generally accepted accounting principles applicable in the circumstances. If these adjustments had been made, reported net income for the period November 29, 1961 to November 30, 1962 would have been increased $81,000 (decrease in cost of publications sold). The effect on surplus in the accompanying balance sheet is not material.

In our opinion, except as stated in the preceding paragraph, the accompanying statements present fairly the financial position of the American Society for Testing and Materials at November 30, 1962, and the results of operations of the general fund and building fund, and changes in special and designated funds, for the period then ended in conformity with generally accepted accounting principles.

The accompanying supplementary information has been subjected to the tests and other auditing procedures applied in the examination of the financial statements mentioned above and, in our opinion, is fairly stated in all respects material in relation to the financial statements taken as a whole.

Arthur Young & Company

BALANCE SHEET AS OF NOVEMBER 30, 1962

(Including special and designated funds)

ASSETS

General Fund:		
Current:		
Cash		$221 214.82
Investments at Cost (market value $864 220.96)	$765 285.45	
Other Investments at Cost (market value $198 845.50)	198 717.76	964 003.21
Accounts Receivable		142 213.79
Inventories:		
Publications in Process	114 381.73	
Completed Publications	289 469.33	403 851.06
Other Current Assets		17 813.17
Total Current Assets—General Fund		1 749 096.05
Furniture and Fixtures, at Cost Less Accumulated Depreciation	47 651.93	
Company Car, at Cost Less Accumulated Depreciation	2 528.37	50 180.30
Total Assets—General Fund		1 799 276.35
Building Fund:		
Cash		76 018.30
Investments at Cost (market value $99 978.80)		99 775.30
Other Current Assets		11 844.44
Land	330 000.00	
New Headquarters—Construction Costs	201 015.54	531 015.54
Total Assets—Building Fund		718 653.58
Special and Designated Funds:		
Cash		124 820.78
Due from Investment Fund		4 692.78
Contributions Receivable from General Fund		14 900.00
Investments at Cost (market value $311 917.73)		276 209.54
Other Investments at Cost (market value $209 437.63)		177 931.32
Total Assets—Special and Designated Funds		$598 554.42

LIABILITIES

General Fund:			
Current:			
Accounts Payable			$107 444.40
Advance Collections			22 007.89
Due Committees:			
Joint Committee on X-ray Diffraction			315 903.68
International Standards Organization (ISO/TC 17)			4 330.24
Other			1 556.92
Contributions Payable:			
ASTM Research Fund			3 900.00
ASTM Executive Award Fund			11 000.00
Provision for 1962 Proceedings			20 000.00
Dues (1963) Collected in Advance			72 533.12
Total Current Liabilities—General Fund			558 676.25
Reserves:			
Life Membership Fund		$4 816.94	
Book of Standards (1964)		125 000.00	
Executive Retirement		10 473.60	
Retirement Fund		35 881.48	
Special Annual Meeting Account		2 661.64	
		178 833.66	
Surplus		1 061 766.44	1 240 600.10
Total Liabilities and Surplus—General Fund			1 799 276.35
Building Fund:			
Accounts Payable			62 676.38
Surplus			655 977.20
Total Liabilities and Surplus—Building Fund			718 653.58
Special and Designated Funds:			
Committee Funds—Unexpended Balance			434 878.79
Research Fund—Contributions Payable			2 305.68
Special Funds Balances:			
	Principal	*Income*	
ASTM Research Fund	$120 982.15	$1 244.78	122 226.93
Dudley Medal and Marburg Lecture Fund	18 635.95	166.73	18 802.68
Richard L. Templin Award Fund	2 763.49	400.06	3 163.55
Sam Tour Award Fund	2 150.81	145.73	2 296.54
F. E. Richart Award Fund	1 411.26	161.25	1 572.51
Walter C. Voss Award Fund	2 291.40	16.34	2 307.74
ASTM Executive Award Fund	11 000.00		11 000.00
Total Special and Designated Funds			$598 554.42

GENERAL FUND: STATEMENT OF OPERATIONS AND SURPLUS

FOR THE PERIOD NOVEMBER 29, 1961 TO NOVEMBER 30, 1962

Income:			
Members' Dues		$386 318.64	
Entrance Fees		7 765.00	
Publication Sales		1 423 848.95	
Advertising (Net of Discounts)		85 437.79	
Interest and Dividends		34 988.85	
Staff Services		11 834.47	
Gain on Sales of Securities		32 495.42	
Registration Fees—Meetings		51 265.70	
Exhibit		29 454.50	
Postage and Handling		51 404.09	
Miscellaneous		757.99	$2 115 571.40
Expenses:			
Cost of Publications Sold:			
Inventory Beginning		457 699.10	
Publication Costs	$682 915.42		
Provision for Estimated Loss on 1962 Proceedings	20 000.00	702 915.42	
		1 160 614.52	
Inventory Ending		403 851.06	
Cost of Publications Sold		756 763.46	
Administrative Expenses	262 174.00		
Society Office Expenses	915 580.36		
Depreciation	6 870.46		
Bad Debts	2 974.18	1 187 599.00	1 944 362.46
Net Income			171 208.94
Surplus, November 29, 1961		813 850.25	
Restoration of Reserve for 1961 Book of Standards		238 000.00	
Excessive Provision for 1961 Proceedings		8 362.26	
November 28, 1961 Inventory Adjustment		58 669.99	
		1 118 882.50	
Reserve for 1964 Book of Standards	125 000.00		
Cancellation of Loan Receivable from Building Fund	103 325.00	228 325.00	890 557.50
Surplus, November 30, 1962			$1 061 766.44

BUILDING FUND: STATEMENT OF OPERATIONS AND SURPLUS

FOR THE PERIOD NOVEMBER 29, 1961 TO NOVEMBER 30, 1962

Income:			
Rentals:			
General Fund		$75 000.00	
Other		299.25	$75 299.25
Expenses:			
Rent		15 204.00	
Maintenance and Repairs		2 173.25	
Janitorial and Cleaning		7 081.80	
Supplies and Maintenance		1 677.01	
Utilities		8 401.79	
Insurance		528.47	
Taxes		6 188.49	
Moving Expense		4 173.50	
Finance Expense		800.00	46 228.31
Net Income			29 070.94
Surplus, November 28, 1961		351 764.12	
Contributions Received for New Headquarters	$215 365.55		
Less Soliciting Expense	22 297.77	193 067.78	
Cancellation of Loan Payable to General Fund		103 325.00	
		648 156.90	
Adjustment of Carrying Value—Land and Buildings		21 250.64	626 906.26
Surplus, November 30, 1962			$655 977.20

CHANGES IN SPECIAL AND DESIGNATED FUNDS

FOR THE PERIOD NOVEMBER 29, 1961 TO NOVEMBER 30, 1962

ASTM RESEARCH FUND

Principal Account

	Total	*Invested*	*Uninvested*
Balance, November 29, 1961	$112 754 20	$109 004.20	$3 750.00
Transfer from General Fund	3 900.00		3 900.00
Transfer from Income—Gain on Investments	4 327.95	4 327 95	
Additional Investment		3 750.00	(3 750.00)
Balance, November 30, 1962	$120 982.15	117 082.15	3 900.00
Investments	$107 007.52		
Other Investments, at Cost (market value $39 978.25)			
618 shares General Motors Co Com (Note A)	7 258.63		
48 shares American Telephone & Telegraph (Note A)	2 816.00	$117 082.15	
Due from General Fund			$3 900.00

Income Account

Balance, November 28, 1961	$6 846.53	
Receipts: Interest, Dividends and Gain on Investments	7 846.12	$14 692.65
Disbursements:		
Transfer to Principal	4 327.95	
Printing	15.25	
Research:		
Engineering Thesaurus	500.00	
University of Delaware	1 104.67	
Corrosion Research Council	500.00	
Battelle Memorial Institute	3 000.00	
Damping Properties of Materials	1 000.00	
Elastic Plastic Behavior of Metals	1 000.00	
Thermoelectricity	1 000.00	
Fatigue Performance of Materials	1 000.00	13 447.87
Balance, November 30, 1962		1 244.78
Composed of:		
Cash	1 801.71	
Due from Investment Fund	1 748.75	
Contributions Payable	(2 305.68)	$1 244.78

NOTE A.—The income from these securities is paid to annuitants.

DUDLEY MEDAL AND MARBURG LECTURE FUND

Principal Account

	Invested	*Uninvested*
Balance, November 29, 1961	$8 273.98	$10 000.00
Transfer from Income—Gain on Investments	361.97	
Invested	10 000.00	(10 000.00)
Balance, November 30, 1962	18 635.95	
Investment Fund Participation	18 635.95	

Income Account

Balance, November 29, 1961—Cash	160.05	
Receipts: Interest, Dividends and Gain on Investments	993.37	1 153.42
Disbursements:		
Transfer to Principal	361.97	
Dudley Medals	109.12	
Marburg Award	500.00	
Marburg Certificates	15.60	986.69
Balance, November 30, 1962		166.73
Composed of:		
Due from Investment Fund	483.42	
Cash	(316.69)	$166.73

RICHARD L. TEMPLIN AWARD FUND

Principal Account

Balance, November 29, 1961—Invested		$2 647.66
Transfer from Income—Gain on Investments		115.83
Balance, November 30, 1962—Invested		2 763.49

Income Account

Balance, November 29, 1961—Cash	$380.69	
Receipts: Interest, Dividends and Gain on Investments	208.34	589.03
Disbursements:		
Transfer to Principal	115.83	
Awards	50.00	
Certificates	23.14	188.97
Balance, November 30, 1962		400.06
Composed of:		
Cash	354.90	
Due from Investment Fund	45.16	$400.06

SAM TOUR AWARD FUND

Principal Account

Balance, November 29, 1961—Invested		$2 060.66
Transfer from Income—Gain on Investments		90.15
Balance, November 30, 1962—Invested		2 150.81

Income Account

Balance, November 29, 1961—Cash	$234.73	
Receipts: Interest, Dividends and Gain on Investments	162.16	396.89
Disbursements:		
Transfer to Principal	90.15	
Awards	135.27	
Certificates	25.74	251.16
Balance, November 30, 1962		145.73
Composed of:		
Cash	110.58	
Due from Investment Fund	35.15	$145.73

F. E. RICHART AWARD FUND

Principal Account

Balance, November 29, 1961—Invested		$1 352.11
Transfer from Income—Gain on Investments		59.15
Balance, November 30, 1962—Invested		1 411.26

Income Account

Balance, November 29, 1961—Cash	$114.01	
Receipts: Interest, Dividends and Gain on Investments	106.39	220.40
Disbursements: Transfer to Principal		59.15
Balance, November 30, 1962		161.25
Composed of:		
Cash	138.19	
Due from Investment Fund	23.06	$161.25

WALTER C. VOSS AWARD FUND

Principal Account

	Total	*Invested*	*Uninvested Cash*
Balance, November 29, 1961	None	None	None
Contribution from W. H. Gompertz	$2 500.00		$2 500.00
Award	(193.00)		(193.00)
Certificate	(15.60)		(15.60)
Invested		$1 000.00	(1 000.00)
Balance, November 30, 1962	$2 291.40	$1 000.00	$1 291.40

Income Account

Balance, November 29, 1961	None
Receipts: Interest and Dividends on Investments	16.34
Balance, November 30, 1962—Due from Investment Fund	$16.34

ASTM EXECUTIVE AWARD FUND

Principal Account

Balance, November 29, 1961	None
Transfer from General Fund	$11 000.00
Balance, November 30, 1962—Due from General Fund	$11 000.00

SPECIAL AND DESIGNATED FUNDS

AS OF NOVEMBER 30, 1962

COMMITTEE FUNDS

Committee A-1	On steel	$2 951.45
Committee A-5	On corrosion of iron and steel	2 629.39
Committee B-3	On corrosion of nonferrous metals and alloys	2 461.71
Committee B-4	On metallic materials for thermostats and for electrical heating, resistance, and contacts	74.74
Committee B-5	On copper and copper alloys, cast and wrought	1.21
Committee B-6	On die-cast metals and alloys	3 876.89
Committee B-8	On electrodeposited metallic coatings and related finishes	40.00
Committee C-1	On cementitious, ceramic, concrete, and masonry materials:	
	General funds	1 645.81
	Cement reference laboratory	(274.67)
	Cement reference laboratory—inspection	9 040.06
Committee C-7	On lime	8.92
Committee C-9	On concrete and concrete aggregates	219.35
	Sanford E. Thompson Medal Award	839.23
Committee C-14	On glass and glass products	58.17
Committee C-15	On manufactured masonry units	651.56
Committee C-16	On thermal insulating materials	223.41
Committee C-17	On asbestos-cement products	57.83
Committee C-18	On natural building stones	151.61
Committee D-1	On paint, varnish, lacquer, and related products	1 673.46
Committee D-2	On petroleum products and lubricants	3 481.71
	ASTM—IBM	215.03
	DCC I Reference fuel account	50 941.96
Committee D-3	DCC II National exchange group	(1 200.81)
Committee D-4	On road and paving materials	1 069.81
Committee D-5	On coal and coke	1 816.60
Committee D-7	On wood	64.40
Committee D-9	On electrical insulating materials	2 501.26
Committee D-10	On shipping containers	0

Committee D-11	On rubber and rubber-like materials		2 745.28
Committee D-12	On soaps and other detergents		1 425.61
Committee D-13	On textile materials		3 155.62
Committee D-14	On adhesives		67.65
Committee D-15	On engine antifreezes		25.87
Committee D-18	On soils for engineering purposes		102.47
Committee D-19	On industrial water		252.99
	On research project		236.26
Committee D-20	On plastics		6 314.53
Committee D-27	On electrical insulating liquids and gases		1 108.29
Committee E-2	On emission spectroscopy		570.02
Committee E-3	On chemical analysis of metals		219.56
Committee E-4	On metallography		46.57
Committee E-7	On nondestructive testing		7 671.20
Committee E-9	On fatigue		308.94
Committee E-13	On absorption spectroscopy		2 579.63
Committee E-14	On mass spectrometry		5 122.90
Committee E-19	On gas chromatography		7 327.62
Committee F-1	On materials for electron tubes and semiconductor devices		591.60
ASA sectional committee on specifications for cast iron pipe and fittings—general fund			1 777.01
Central New York district educational fund			958.32
Chicago district			2 167.74
Cleveland district			36.37
Detroit district			2 100.28
Mississippi Valley			352.93
New England district			2 835.01
New York district			10.06
New York district special			8 062.51
Northern California district			4 656.43
Ohio Valley district			203.98
Pacific Northwest district			605.51
Philadelphia district			581.67
Philadelphia district special (annual meeting account)			1 605.74
Pittsburgh district			1 698.69
Southern California district			5 202.16
Southern California district special			309.02
Southwest district			174.97
Washington district (Middle Atlantic)			514.79
Western New York - Ontario district			1 480.35
Advisory committee on corrosion			26 196.61
Joint committee on X-ray powder diffraction:			
Chemical analysis			226 138.17
Research fellowship			14 251.61
Joint committee on leather			505.50
Reserve—future West Coast meetings			7 360.66
			434 878.79
Accounted for as follows:			
Cash in bank and on hand		$121 440.69	
Due from investment fund		2 340.90	
Investments at cost (market value $168 459.38):			
$20 000 US Treasury 2½s due 8/15/63		19 925.00	
70 000 Federal Land Banks 4¼s due 7/20/66		70 939.50	
75 000 US Treasury 4⅝ per cent due 5/15/65		76 992.19	
Investment fund participation		143 240.51	$434 878.79

ASTM INVESTMENT FUND

As of November 30, 1962

Cash	$41 835.45	
Investments, at Cost (market value $1 151 349.77)	1 016 673.51	
Interest and Dividends Receivable	7 116.35	
Total Assets		$1 065 625.31
Participations by Funds	1 041 494.99	
Due to Participating Funds	17 020.46	
Undistributed Amounts Due Participating Funds:		
Accrued Interest and Dividends	7 116.35	
Loss on Sale of Investment	(6.49)	$1 065 625.31

Participation by Funds

	Participation Nov. 28, 1961	*Additional Cash Transferred*	*Net Profit Sale of Securities Reinvested*	*Participation Nov. 30, 1962*
General Fund	$708 887.66	$23 902.37	$32 495.42	$765 285.45
Committee Funds	137 236.71		6 003.80	143 240.51
Research Fund	98 929.57	3 750.00	4 327.95	107 007.52
Building Fund	33 902.37	(33 902.37)		
Medal and Lecture Fund	8 273.98	10 000.00	361.97	18 635.95
Templin Award Fund	2 647.66		115.83	2 763.49
Sam Tour Fund	2 060.66		90.15	2 150.81
Richart Fund	1 352.11		59.15	1 411.26
Walter C. Voss Fund		1 000.00		1 000.00
	$993 290.72	$4 750.00	$43 454.27	$1 041 494.99

ASTM INVESTMENT FUND: STATEMENT OF INCOME

For the Period November 29, 1961 to November 30, 1962

Income:		
Interest and Dividends on Investments	$39 285.89	
Gain on Sales of Investments	43 447.78	$82 733.67
Expenses:		
Advisory Fees	1 750.00	
Custodian Fees	2 572.58	
Postage	1.50	
Commission—Short Term	4.00	
Affidavit Canadian Tax	0.75	4 328.83
Net Income		78 404.84

	Interest and Dividends	*Capital Gain*	
Distribution to Participants (Note):			
General Fund	$25 612.97	$32 495.42	
Committee Funds	4 795.47	6 003.80	
ASTM Research Fund	3 518.17	4 327.95	
Dudley Medal and Marburg Lecture Fund	631.40	361.97	
Templin Award Fund	92.51	115.83	
Sam Tour Award Fund	72.01	90.15	
F. E. Richart Award Fund	47.24	59.15	
Walter C. Voss Award Fund	16.34		
	34 786.11	43 454.27	78 240.38
Excess of Net Income over Amounts Distributed			164.46
Composed of:			
Increase in Accrued Interest and Dividends		170.95	
Undistributed Capital Loss		(6.49)	$164.46

NOTE.—Includes $17 020.46 interest and dividends to be paid to participating funds January, 1963.

ADMINISTRATIVE EXPENSES

For Period November 29, 1961 to November 30, 1962

Board of Directors' Meetings	$14 228.80
Special and Administrative Travel	7 073.36
Circular Letters and Notices to Members	10 209.86
President's Office	3 000.00
Educational Grants	11 500.00
Retirement	51 465.02
Social Security	14 442.43
Group Insurance	3 840.24
Major Medical	3 042.11
Blue Cross	4 901.26
Legal, Actuarial and Related	15 936.89
American Standards Association	2 250.00
Engineers Joint Council	823.38
Special Contributions	2 525.00
Contribution to Research Fund	3 900.00
Contribution to Executive Award Fund	11 000.00
Annual Meeting	19 306.62
Committee Week (Spring)	6 663.65
National Meetings	24 358.17
Other Meetings	2 944.24
District Activities	5 083.34
Technical Committees	14 014.48
Honors and Awards	4 563.64
Membership Certificates, Pins	320.09
Photographic Exhibit	2 425.10
Apparatus Exhibit	17 310.81
Washington Representative	5 045.51
	$262 174.00

SOCIETY OFFICE EXPENSES

For Period November 29, 1961 to November 30, 1962

Salaries	$610 783.94
Commissions	12 946.95
Postage	39 601.89
Duplicating and Printing	34 847.90
Travel	22 652.89
Telephone and Telegraph	15 384.52
Addressograph Service	4 016.92
Insurance	1 092.63
Service Contracts	8 078.60
Secretary's Emergency Fund	251.58
Collection and Exchange	238.71
Mailing Service	8 915.73
Publication Storage	28 167.64
Stationery and Supplies	32 041.46
Rent	75 000.00
Subscriptions and Dues	1 912.89
Advertising Promotion	3 435.69
Audit Bureau of Circulation	1 659.97
Univac (Payroll Preparation)	848.45
Auto Expense	58.26
Employment Costs	1 683.68
Exhibiting Charges and Shipping	458.30
Division of Materials	144.67
Miscellaneous	11 357.09
	$915 580.36

APPENDIX II

REPORT OF ADMINISTRATIVE COMMITTEE ON STANDARDS

So that the purposes of the Society can be carried out effectively, it is necessary that the technical committees keep abreast of changes in technology and that the Society provide a means of reflecting these changes promptly in its standards. This was one of the reasons for the organization of the Administrative Committee on Standards in 1929. Until that time, the Annual Meeting was the place where all actions on standards and tentatives were transacted. It was recognized that a means should be provided for actions by the Society between Annual Meetings on tentatives, and the Administrative Committee on Standards was accordingly organized, with one of its functions to act for the Society between Annual Meetings on new and revised tentatives, tentative revisions of standards, and the withdrawal of tentatives and standards. The technical committees have made very extensive use of this interim procedure, as shown below. The democratic principles on which the Society is based have in no way been sacrificed by investing authority in the administrative committee. In fact, these principles have been enhanced by the very thorough review given to the vote in the technical committees and the information on consideration given by the committees to any negative votes to determine whether the recommendations represent a true consensus. The committee report sessions at the Annual Meeting have at times been lacking in this regard and the Standards Committee is very anxious to see that the character of these sesssions is improved. During the 1962 Annual Meeting, two or more members of the Standards Committee monitored report sessions, and when a committee report or ballot did not appear to be in accordance with Society Regulations it was questioned and appropriate action taken.

Another milestone was reached in September, 1962, when the Society approved a change in its By-laws providing for periodic action by the Society on standards as well as tentatives in the interval between Annual Meetings. The main reason for this change is the fact that in many areas changes in technology are occurring very rapidly and it is often not feasible to hold the new or revised standards for Annual Meeting action. Some committees, although recognizing that documents deserved standard status, have kept them as tentative since they wished to be able to make revisions as needed. The new provision for interim action on standards will enable the committees to make a more realistic appraisal of the tentatives and accordingly adopt many as standard. In certain circles outside ASTM, tentatives are no longer considered as having official status, and the issuance of more documents as standard will alleviate the difficulties encountered by these groups.

The Administrative Committee on Standards is prepared to assume the additional responsibility in acting on standards. Changes in the Regulations Governing Technical Committees to provide a procedure to be followed in connection with these actions on standards are being worked out by an ad hoc committee appointed by the Board of Directors. These changes were presented to the Board for final action at its May, 1963, meeting.

With the increased role of the Standards Committee in the standardization activities of the Society, there must be a thorough understanding on the part of the Society's members and committee members as to its functions. The Standards Committee has found that there is a need for a better understanding by the technical committees as to what is required for a proposal to become an ASTM standard and possibly later an American Standard. In this connection a paper has been prepared by the chairman entitled "The Administrative Committee on Standards—What It Is and What It Does." It is planned to publish the paper in an early issue of *Materials Research & Standards*. Reprints or excerpts of the paper will be made available for distribution to all committee members.

The following individuals comprise the present membership of the Administrative Committee on Standards:

G. H. Harnden (Chairman), General Electric Co.
R. M. Berg, Union Carbide Chemicals Co.
L. J. Jacobi (Retired), The Detroit Edison Co.
W. A. Kirklin, Hercules Powder Co.
J. L. Menson, Combustion Engineering, Inc.
H. C. Miller, Public Service Electric and Gas Co.
M. A. Pinney, The Pennsylvania Railroad Co.
J. B. Rather, Jr., Socony Mobil Oil Co.
F. W. Reinhart, Consultant
I. V. Williams, Bell Telephone Laboratories, Inc.

For a number of years R. E. Hess, in his capacity as Technical Secretary of the Society, served as secretary of the Standards Committee. In January, 1963, he was succeeded as Technical Secretary by J. W. Caum and at that time Miss Betty J. Preston, who had assisted Mr. Hess in the secretarial work for the past 17 years was appointed secretary of the Standards Committee. The committee wishes to record its appreciation of the many valuable contributions made by Mr. Hess to this work over the years and its appreciation of him as an individual.

In addition to its responsibility for acting for the Society on standards and tentatives as indicated above, the Administrative Committee on Standards is responsible to the Board of Directors for the general supervision of the standardization activities of the Society in the following areas: (1) promotion and expansion of standardization work in new fields, (2) formulation of a general policy concerning standardization activities, and (3) review of the Society's standardization work. These activities are described later in this report, but the more important actions taken in the several categories are referred to briefly here. In the area of promotion and expansion of standardization, the administrative committee took action on the following: (1) space simulation, (2) pyrolytic materials, (3) activated carbon, (4) structural carbon, (5) temperature measurement, (6) control of contaminants, (7) thermal radiation, (8) rock mechanics, and (9) peat, peat moss, and peat humus products.

The Standards Committee has given or is giving serious consideration to several matters having to do with general policy concerning standardization activities:

1. Reservation of blocks of designations for standards in specific fields,
2. Change in system for designations of ASTM standards,
3. Review of Recommendations on Form of ASTM Standards,
4. Point system for numbering sections of ASTM standards, and

5. Changes in Regulations Governing Technical Committees.

Actions by the Society at 1962 Annual Meeting:

In the area of review of the Society's standardization work, the administrative committee took cognizance of the recommendations affecting standards accepted by the Society at the 1962 Annual Meeting. The various actions reported are:

New Standards and Existing Tentatives Adopted as Standard	118
Standards in Which Revisions Were Adopted	117
New Tentatives	110
Standards Revised and Reverted to Tentative	15
Tentative Revisions of Standards	11
Tentatives Revised	139
Standards and Tentatives Withdrawn	31

Actions by the Administrative Committee on Standards During the Year:

A number of the technical committees are taking advantage of the procedure for action on recommendations between Annual Meetings through the Administrative Committee on Standards. During the past year, 33 technical committees submitted a total of 217 proposals for review and action as follows. The details of these recommendations have been reported in *Materials Research & Standards.*

New Tentatives	72
Revisions of Tentatives	108
Standards Revised and Reverted to Tentative	31
Tentative Revisions of Standards	2
Tentatives Withdrawn	1
Standards Withdrawn	3

In addition, reports from six sectional committees, for which ASTM is sponsor or joint sponsor, were reviewed as referred to in a later section of this report.

The Administrative Committee on Standards carries on the bulk of its activities through correspondence. Since the last Annual Meeting of the Society, the committee held two meetings: on Oct. 17, 1962, and on April 10–11, 1963.

EXPANSION OF STANDARDIZATION ACTIVITIES

A number of new standardization projects are discussed below:

Space Simulation.—The request of a number of companies concerned with the space program that work be undertaken by ASTM on space simulation was explored at a general conference held on Oct. 30, 1962, in Los Angeles, Calif. The conference was unanimous in recommending the formation of a new ASTM committee on space simulation which was authorized subsequently by the Board and assigned the designation E-21. In the organization of this committee the Society has the full cooperation of other interested societies, the National Aeronautics and Space Administration, the U. S. Government, and companies such as the Aerospace Corp. The new committee also has the support of the other technical committees of the Society concerned with this work. The organization meeting was held on April 15, 1963, in Los Angeles, Calif.

Pyrolytic Materials.—Because of the increasing use of pyrolytic materials, especially in the aerospace industry, and the need for recognized standards for these materials, a conference held on Nov. 9, 1962, recommended the formation of a new ASTM Committee on Pyrolytic Materials. This recommendation received approval by the Board on Jan. 15, 1963, and the new committee has been assigned the designation C-6. The organization meeting was held on Feb. 25, 1963. The Standards Committee has recommended that the definition of pyrolytic materials be added as a note to the scope since the term does not seem to be in general use.

Activated Carbon.—A number of producers and consumers of activated carbon met on June 19, 1962, to explore the possibility of ASTM undertaking the work needed on activated carbon. The three major uses are for water purification, sugar decolorization, and gas-phase reactions. No existing committee of the Society was prepared to handle such work and the conference recommended the formation of a new ASTM committee. The Board authorized the new committee (D-28) and the organization meeting was held on Nov. 15, 1962, in Philadelphia, Pa.

Structural Graphite.—Numerous rapid developments in the field of structural graphite prompted Committee C-21 on Ceramic Whitewares and Related Products, which was then at work on nuclear graphite and graphite pipe, to recommend the formation of a new ASTM Committee on Structural Graphite. This request was granted by the Board in Sept., 1962, and the organization meeting of the new committee (C-5) was held on May 21-22, 1963, in Cleveland, Ohio.

Temperature Measurement.—Subcommittees 32 on Thermocouples and 17 on Thermometers have been removed from Committee E-1 on Methods of Testing to serve as the nucleus for the new Committee E-20 on Temperature Measurement. The organization meeting of the committee was held on Dec. 13, 1962.

Peat, Peat Moss, and Peat Humus Products.—The Peat Producers Assn. has requested that ASTM prepare definitions and methods of test for the physical properties of peat and peat products to include acidity, pH, moisture content, organic content, weight per cubic foot, consistency, moisture holding capacity, and the ability to support plant life. Such standardization of the methods would enable the producer to indicate on the packages of peat an analysis of the material, as is now done with fertilizers. The standardized definitions would permit accurate description of the various peat deposits. The Standards Committee has recommended the formation of a new ASTM committee to deal with the subject. The project originally appeared to be one for the Association of Official Agricultural Chemists or Horticultural Society, but these groups feel it is outside their scopes and have offered their support to an ASTM committee.

Agricultural Products.—The cooperation of ASTM has been requested in the preparation of methods to determine certain properties of grain and other agricultural products which are needed in connection with the design of machines used in processing these products. The Standards Committee has recommended that a conference be called to explore this matter.

Rock Mechanics.—The Standards Committee has recommended that the request for ASTM to undertake work on rock mechanics be referred to Committee D-18 on Soils for Engineering Purposes, since such work is closely related to soil mechanics.

Control of Contaminants.—A number of committees of the Society are concerned with the control of contaminants —not only the control of airborne particulate contamination of clean rooms, but also the cleanliness of liquids and gases for processing and the cleanliness of surfaces. Coordination of this work is needed and the Standards Committee has recommended that a conference be held to plan the most effective means within the Society for standardization in this field.

Thermal Radiation.—The Standards Committee has recommended that serious consideration be given by Committee E-21 on Space Simulation to a request for that committee to carry out work on thermal radiation. The request

ASTM PARTICIPATION IN INTERNATIONAL STANDARDS

Committee	Title	Secretariat	Responsible Group	Sponsor of USA Activity	ASTM Participation Through:
ISO (INTERNATIONAL ORGANIZATION FOR STANDARDIZATION)					
TC6	Paper	France	USA National Committee	ASA	D-6
TC8	Shipbuilding Details	Netherlands	USA National Committee	ASA	B-7
TC17	Steel	Great Britain	USA National Committee	ASTM	A-1
TC18	Zinc and Zinc Alloys	Belgium	(Observer status)	ASTM	B-2
TC24	Sieves	Germany	ASA Z23	ASTM, NBS	E-1
TC25	Cast Iron	Great Britain	USA National Committee	ASTM	A-3, A-7
TC26	Copper and Copper Alloys	Germany	USA National Committee	ASTM	B-5, B-2
TC27	Solid Mineral Fuels	Great Britain	USA National Committee	ASTM	D-5 Subcommittee XXVII
TC28	Petroleum Products	USA	ASA Z11	ASTM	D-2
TC33	Refractories	Great Britain	USA National Committee	ASTM	C-8
TC35	Raw Materials for Paints, Varnishes, and Similar Products	Netherlands	USA National Committee	ASTM	D-1
TC38	Textiles	Great Britain	USA National Committee L23	ASTM, AATCC	D-13
TC45	Rubber	Great Britain	USA National Committee	ASTM	D-11
TC47	Chemistry	Italy	USA National Committee	ASTM	E-15
TC48	Laboratory Glassware and Related Apparatus	Great Britain	USA National Committee	ASA	E-1
TC50	Lac	India	USA National Committee	ASTM	D-1 Subcommittee XI
TC56	Mica	India	USA National Committee	ASTM	D-9 Subcommittee IX
TC61	Plastics	USA	USA National Committee	ASTM	D-20 Subcommittee XXI
TC66	Viscosity	USA	USA National Committee	ASTM	E-1 Subcommittee 9
TC69	Statistical Treatment of Series of Observations	Netherlands	USA National Committee	ASA	E-11
TC74	Hydraulic Binders	Belgium	ASA A1	ASTM	C-1
TC77	Products in Asbestos Cement	Switzerland	USA National Committee	ASTM	C-17
TC78	Aromatic Hydrocarbons	Great Britain	USA National Committee	ASTM	D-16 Subcommittee H
TC79	Light Metals and Their Alloys	France	USA National Committee	ASTM	B-7
TC89	Derived Timber Products	Germany	(Observer status)	ASTM	D-7
TC91	Surface Active Agents	France	In process	...	D-12 interest
TC92	Fire Tests on Building Materials & Structures	Great Britain	(Observer status)	ASTM	E-5
TC102	Iron Ores	Japan	USA National Committee	ASTM	E-16
TC107	Treatments of Metallic Surfaces	Italy	In process	...	A-5 and B-8 interest

IEC (INTERNATIONAL ELECTROTECHNICAL COMMISSION)

TC7	Aluminum	...	US National Committee IEC	...	ASA C7 (B-1)
TC10	Insulating Oils	...	US National Committee IEC	...	ASA C59 (D-27)
TC14A	Magnetic Steel	...	US National Committee IEC	...	A-6
TC15	Insulating Materials	...	US National Committee IEC	...	ASA C59 (D-9)

CPNT (PAN AMERICAN STANDARDS COMMISSION)

TC2	Steel	Chile	US National Committee	ASTM	A-1
	Building Materials	...	...	...	C-1
	Textiles	Peru	US National Committee	ASTM	D-13
TC8	Cattle Products	Argentina	US National Committee	ASTM, ALCS	Joint Committee on Leather

is being made by a group which heretofore has been unaffiliated with an established society.

RELATIONS WITH AMERICAN STANDARDS ASSN.

Two methods under the procedure of the American Standards Assn. are used by ASTM in submitting standards to the ASA. One is a combination of the so-called existing standards procedure and the proprietary sponsorship procedure. On the initial reference of an existing ASTM standard to the ASA, it is submitted under the existing standards procedure with a supporting statement giving a history of the development of the standard and indicating the degree of its acceptance. If the standard is approved as American Standard, the ASTM is granted proprietary sponsorship for future revisions, and such revisions are brought to the attention of the ASA currently for approval under the proprietary sponsorship procedure.

The second method consists of having standards reviewed in a sectional committee made up of representatives of a number of interested organizations, usually with the ASTM as sponsor or co-sponsor of the sectional committee.

Standards Submitted Under the Existing Standards Procedure:

The following standards have been submitted to the American Standards Assn. during the year for approval as American Standard under the Existing Standards Procedure: 4 standards relating to steel, 11 standards relating to copper and copper alloys, 32 relating to thermal insulating materials, 3 relating to gaseous fuels, 21 relating to rubber and rubber-like materials, 2 relating to engine antifreezes, and 10 relating to general laboratory apparatus and methods of testing.

Revised Standards Submitted Under the Proprietary Procedure:

In view of the Society having been designated as proprietary sponsor for the revision of ASTM standards that had been approved as American Standard under the existing standards procedure, it has submitted to the American Standards Assn. during the year revisions of 7 standards relating to steel, 4 standards relating to copper and copper alloys, 1 relating to lime, 2 relating to gypsum, 2 relating to refractories, 7 to manufactured masonry units, 2 to rubber, and 1 to thermometers.

Standards Submitted Under the Sectional Committee Procedure:

During the year the following sectional committees, for which the Society is sponsor or joint sponsor, took action to present recommendations to the ASA as follows:

Sectional Committee A1 on Specifications and Methods of Test for Hydraulic Cement.—This committee recommended the approval as American Standard of 2 standards and the approval of revisions of 3 American Standards.

Sectional Committee B36 on Dimensions and Materials of Wrought-Iron and Wrought-Steel Pipe and Tubing.—This committee recommended the approval as American Standard of 4 standards, the approval of revisions of 29 American Standards, and the withdrawal of 1 American Standard.

Sectional Committee C7 on Bare Electrical Conductors.—This committee recommended the approval as American Standard of 1 standard, the approval of revisions of 5 American Standards, the withdrawal of 2 American Standards, and the reaffirmation of 4 American Standards.

Sectional Committee C59 on Electrical Insulating Materials.—This committee has recommended the approval as American Standard of 11 standards, the approval of revisions of 1 American Standard, and the reaffirmation of 5 American Standards.

Sectional Committee L14 on Textile Test Methods.—This committee has recommended the approval as American Standard of 18 standards, the approval of revisions of 21 American Standards, and the withdrawal of 3 American Standards.

Sectional Committee Z11 on Petroleum Products and Lubricants.—This committee has recommended the approval as American Standard of 7 standards, the approval of revisions of 11 American Standards, and the withdrawal of 2 American Standards.

INTERNATIONAL RELATIONS

The international discussions, particularly under the International Organization for Standardization (ISO) and the International Electrotechnical Commission (IEC), and more recently under the Pan American Standards Committee, are assuming ever greater significance. A number of the European and South American countries apparently wish to have standardization discussions handled internationally, and much of the work is supported here rather than in the development of individual national standards. In certain areas these international discussions are having a definite impact on the standardization in this country, and a number of ASTM committees are following these international discussions quite closely with the thought that the ASTM standards and the international recommendations should be in harmony. A list of the various ISO, IEC, and Pan American projects in which ASTM is interested and which continues to grow year by year is given in the accompanying table. Reports appear currently in *Materials Research & Standards* in connection with meetings of the committees responsible, which refer to the actions taken and to the delegations that attend these meetings.

Respectfully submitted on behalf of the committee,

G. H. HARNDEN,
Chairman.

BETTY J. PRESTON,
Secretary.

APPENDIX III

REPORT OF ADMINISTRATIVE COMMITTEE ON RESEARCH

During 1962, the Administrative Committee on Research met twice—on June 24 in New York City and on Nov. 18 in Philadelphia.

There are now ten members of the committee, including the secretary and two ex officio members from the Board of Directors. A. C. Webber's term as a member expired in June, 1962, and as President of the Society, he appointed B. W. Gonser and I. V. Williams to represent the Board. H. M. Smith, Chairman of Committee D-2 on Petroleum Products and Lubricants, was appointed as a new committee member.

The Research Fund has continued its growth and now amounts to approximately $121,000. Disbursements from the income account totaled about $19,000, including $2500 for the continuing project on absorption spectroscopy, $3000 for work on materials reviews at Battelle Memorial Inst., and $500 for cooperation with the Engineers Joint Council in development of an engineering thesaurus. In addition, research grants were made to University of Delaware, $1200; Purdue University, $1000; University of Minnesota, $1000; William Marsh Rice University, $1000; and Rensselaer Polytechnic Inst., $1000. The amount of $7400 was transferred to the principal account of the Research Fund.

The committee has continued its practice of reviewing research projects in which ASTM has participation or direct interest.

Members of the committee have contributed regularly to the ACR Notes column which appears in *Materials Research & Standards*. It is intended that these short items will serve both to acquaint the Society's membership with the activities of ACR and to encourage interest in the research aspects of the Society's work.

Since it is an important purpose of the Administrative Committee on Research to stimulate interest in materials research and to promote knowledge of materials, various projects are undertaken to further these objectives. During the past year, the following progress has been made:

1. The technical committees have been canvassed to determine the degree of interest in informal research forums. All replies were favorable. It is hoped that a beginning can be made toward implementing this idea at a national meeting in the near future.

2. The ASTM Lecture on Outstanding Research was established with joint sponsorship of the ASTM Board of Directors, the Division of Materials Sciences, and the Administrative Committee on Research. The lecture carries an honorarium furnished from the Research Fund. Two lecturers were selected for the first year: Cyril Stanley Smith, Institute Professor, Massachusetts Institute of Technology, who spoke at Montreal in February, 1963, and Norman Percy Allen, National Physical Laboratory, England, who will speak

at New York City in August, 1963, on the occasion of the International Conference on Creep.

3. "Materials Reviews" was established as a regular department feature in *Materials Research & Standards* under the sponsorship of the Administrative Committee on Research. The cost of preparing these reviews at Battelle Memorial Inst. is charged to the Research Fund.

Respectfully submitted on behalf of the committee,

WALTER J. SMITH,
Chairman.

FRANK Y. SPEIGHT,
Secretary.

APPENDIX IV

REPORT OF THE ADMINISTRATIVE COMMITTEE ON PAPERS AND PUBLICATIONS

The planning and arranging of programs for meetings has again absorbed much of the attention of the Administrative Committee on Papers and Publications during the past year. The outstanding event in this respect was the Fourth Pacific Area National Meeting. This involved 48 technical sessions at which over 200 papers were presented. The review of these papers alone constituted quite an undertaking, and while many experts in addition to the members of the Papers Committee were called upon, it did involve considerable attention on the part of the Papers Committee members since each individual program committee had on it a member of the Papers Committee in addition to some individual from the West Coast Technical Program Committee. The papers from the Pacific Area meeting are now being edited for publication. While some of the papers will appear in *Materials Research & Standards* and some have been submitted to outside journals, for the most part they will be published in some 15 special technical publications.

Apart from the West Coast meeting and the Annual Meeting, which of itself comprised some 29 sessions, there were several special meetings, including the Symposium on Standards for Filament-Wound Reinforced Plastics, sponsored jointly by the Navy and ASTM Committee D-20 on Plastics; and the Symposium on the Dynamic Behavior of Materials, under the joint auspices of the University of New Mexico and the ASTM Rocky Mountain District. Three meetings are still in prospect—a meeting sponsored by the National Research Council of Canada and ASTM Committee D-18 on Soils for Engineering Purposes, on Laboratory Shear Testing of Soils, to be held in Ottawa in September; a meeting under the joint auspices of the American Nuclear Society and ASTM Committee B-3 on Corrosion of Nonferrous Metals and Alloys, on Corrosion of Zirconium Alloys, to be held in New York in November; and the International Conference on Materials, to be held Feb. 3 to 6, 1964, in Philadelphia.

Two meetings of the Administrative Committee on Papers and Publications were held during the year, one on Nov. 16, 1962, and the other on Jan. 25, 1963.

Messrs. A. C. Smith, R. J. Wirshing, and C. R. Sutton have retired after serving two three-year terms. Messrs. W. K. Anderson, General Electric, A. J. Eickhoff, National Lead Co., and W. E. Chalfant, The Atlantic Refining Co., were appointed to serve a three-year term.

The extent of the Society's publications during the year is presented in the accompanying Record of Publications.

Book of Standards:

The realignment of the Book of Standards beginning in 1964, referred to in the last two annual reports, has received considerable study during the past year. The final plans call for issuing the book annually in 32 parts corresponding roughly to the compilations that have been issued in various areas. Combinations of subjects have been made so as to avoid too great fragmentation and also to secure books of comparable size with no part less than 500 pages. The 6 by 9-in. page size is being maintained, although some consideration has been given to the publication of standards in $8\frac{1}{2}$ by 11-in. size; this latter size will receive further consideration, particularly in connection with the publication of separates. The binding will be the same as that employed on the compilations, which provides a reasonably substantial binding.

Proceedings:

A reconstitution of the *Proceedings* has been under consideration for the past several years. A questionnaire was conducted in the March 1962 issue of *Materials Research & Standards* concerning several possibilities, including splitting the *Proceedings* so that the committee reports would appear in one part and technical papers in another. The study of the various proposals is in the hands of a committee consisting of E. C. Shuman, chairman, A. J. Eickhoff, R. C. Mielenz, and A. Q. Mowbray.

Materials Research & Standards:

The Society's journal *Materials Research & Standards* continues to be well received. It has been possible to provide for somewhat larger issues during the current year. A special advisory committee for *MR&S* has been set up consisting of W. F. Simmons, chairman, Robert Burns, and Bryant Mather.

Thesaurus:

The Administrative Committee on Papers and Publications has been cooperating actively in the discussion that has taken place under the Engineers Joint Council concerning the development of a thesaurus applicable to engineering in general. With such a thesaurus available, authors could be asked to supply key words for indexing of their papers. It is also proposed that an abstract be furnished that would be of assistance to librarians and others interested in indexing and abstracting.

1963 Annual Meeting:

A program of 41 technical sessions is planned for the 1963 Annual Meeting. Of these 27 will be devoted to special symposia. The 10 symposia making up these 27 sessions are listed below:

Symposium on Soil Exploration,
Symposium on Materials Problems in the Design of Supersonic Transport Aircraft,
Symposium on Advances in the Technology of Stainless Steels and Related Alloys,
Symposium on the Chemical and Physical Effects of High Energy Radiation on Inorganic Substances,
Symposium on Space Radiation Effects,
Symposium on Recent Research on Bituminous Materials,
Symposium on Recent Developments in Adhesion Science,
Symposium on Recent Advances in Adhesives Technology,
Symposium on X-ray and Electron Probe Analysis, and
Symposium on Resinographic Methods.

RECORD OF PUBLICATIONS ISSUED
APRIL 1, 1962–APRIL 1, 1963

Regular Publications:

1962 Proceedings, 1350 pp., 4500 copies.
1962 Gillett Lecture, 26 pp., 2500 copies.
1962 Marburg Lecture, 46 pp., 2000 copies.
1962 Supplements to 1961 Book of Standards:
  Part 1, 536 pp., 24,000 copies.
  Part 2, 356 pp., 16,250 copies.
  Part 3, 216 pp., 13,500 copies.
  Part 4, 280 pp., 12,000 copies.
  Part 5, 196 pp., 11,500 copies.
  Part 6, 212 pp., 8500 copies.
  Part 7, 301 pp., 10,500 copies.
  Part 8, 406 pp., 8500 copies.
  Part 9, 304 pp., 9750 copies.

Part 10, 508 pp., 8500 copies.
Part 11, 436 pp., 8000 copies.
1962 Year Book, 820 pp., 9000 copies.
1962 Index to Standards, 240 pp., 25,000 copies.
Materials Research & Standards, 12 issues (April 1962 to March 1963), total number of pages 1070, average number of copies 11,433 (321 pages of technical papers).

A statement of the volume of the principal publications for the past four years is given below:

	1959–1960, pages	1960–1961, pages	1961–1962, pages	1962–1963, pages
Proceedings	1423	1 252	1 470	1 350
Marburg Lecture	32	20	16	46
Gillett Lecture	26	20	18	26
Book of ASTM Standards	....	....	17 356	....
Supplements to Book of Standards	2962	3 172	....	3 751
ASTM Methods of Chemical Analysis of Metals	....	776	....	....
Index to ASTM Standards	224	232	236	240
Year Book	672	792	800	820
* Special Technical Publications	3413	5 060	3 126	3 947
* Other Special Publications	12	34	284	556
Materials Research & Standards	836	856	1 034	1 070
	9600	12 214	24 340	11 806

* Not included under this listing are a number of publications which are partly of the nature of compilations of standards such as:
Building Code Compilation,
Manual of ASTM Standards on Refractory Materials,
Procedure for Testing Soils,
Manual on Industrial Water (STP 148),
Selected ASTM Engineering Materials Standards for Use in College Curricula,
Manual on Measurements and Sampling of Petroleum and Petroleum Products, and
Knock Test Manuals.

Special Compilations of Standards Published from April 1, 1962, to April 1, 1963:

Specifications for Steel Piping Materials (A-1), 526 pp., 2750 copies.
ASTM Standards on Cast Iron (A-3), 240 pp., 1250 copies.
ASTM Standards on Metallic Electrical Conductors (B-1), 400 pp., 750 copies.
ASTM Standards on Copper and Copper Alloys (B-5), 808 pp., 1200 copies.
ASTM Standards on Light Metals and Alloys (B-7), 420 pp., 1000 copies.
ASTM Standards on Metal Powders and Metal Powder Products (B-9), 116 pp., 1200 copies.
ASTM Standards on Cement (C-1), 310 pp., 2000 copies.
ASTM Standards on Mineral Aggregates, Concrete, and Nonbituminous Highway Materials (C-9, D-4), 496 pp., 3000 copies.
ASTM Standards on Glass and Glass Products (C-14), 182 pp., 1000 copies.
ASTM Standards on Thermal Insulating Materials (C-16), 282 pp., 1500 copies.
ASTM Standards on Paint, Varnish, Lacquer and Related Products (D-1), 1554 pp., 2000 copies.
ASTM Standards on Petroleum Products and Lubricants (1961), Part II, 788 pp., 3500 copies.
ASTM Standards on Petroleum Products and Lubricants (1962), Part 1, 1338 pp., 7000 copies.
ASTM Standards on Petroleum Products and Lubricants (1962), Part II, 758 pp., 3000 copies.
ASTM Standards on Gaseous Fuels (D-3), 374 pp., 1200 copies.
ASTM Standards on Bituminous Materials for Highway Constructions, Waterproofing and Roofing (D-4, D-8), 568 pp., 2500 copies.
ASTM Standards on Coal and Coke (D-5), 212 pp., 1500 copies.
ASTM Standards on Textile Materials (D-13), 1028 pp., 2000 copies.
ASTM Standards on Adhesives (D-14), 384 pp., 1500 copies.
ASTM Standards on Benzene, Toluene, Xylene and Solvent Naphtha (D-16), 228 pp., 2000 copies.
Manual on Industrial Water and Industrial Waste Water (D-19) (STP 148-F), 774 pp., 2000 copies.
ASTM Standards on Plastics (D-20), 1484 pp., 2500 copies.
ASTM Methods of Atmospheric Sampling and Analysis (D-22), 154 pp., 1500 copies.
ASTM Standards on Electrical Insulating Liquids and Gases (D-27), 478 pp., 1000 copies.
ASTM Standards on Electron Tube Materials (F-1), 404 pp., 1000 copies.

Special Compilations of Standards in Prospect:

Manual of ASTM Standards on Refractory Materials (C-8), 268 pp., 1500 copies.
Building Code Compilation, 1305 pp., 2500 copies.

Special Publications Issued from April 1, 1962, to April 1, 1963:

ASTM-IP Petroleum Measurement Tables (American Edition), 556 pp., 1500 copies.
1960 References on Fatigue (STP 9-L), 63 pp., 1000 copies.
ASTM Manual on Quality Control of Materials (STP 15-C), 152 pp., 3000 copies.
Index to the X-Ray Powder Data File (STP 48-L), 1054 pp., 1500 copies.
X-Ray Powder Data Cards, Set 12—Inorganic 500 cards, 1700 copies; organic 300 cards, 900 copies.
Supplement to Bibliography and Abstracts on Electrical Contacts (STP 56-P), 54 pp., 1200 copies.
Supplement to Bibliography and Abstracts on Thermostat Metals (STP 288-A), 19 pp., 1000 copies.
Symposium on Non-Newtonian Viscometry (STP 299), 161 pp., 2000 copies.
Symposium on Evaluation of Metallic Materials in Design for Low-Temperature Service (STP 302), 251 pp., 2500 copies.
Symposium on Elevated-Temperature Compression Testing of Sheet Materials (STP 303), 90 pp., 2000 copies.
Symposium on Radiation Effects in Refractory Fuel Compounds (STP 306), 201 pp., 2500 copies.
Symposium on Extension of Sensitivity for Determining Various Constituents in Metals (STP 308) 74 pp., 2000 copies.
Papers on Road and Paving Materials and Symposium on Microviscometry (STP 309), 155 pp., 2000 copies.
Handbook on Vapor Degreasing (STP 310), 39 pp., 1500 copies.
Evaluation of Compression Set Testing—Standard Versus Variable Deflection Methods, by William H. King and R. E. Harding (STP 311), 31 pp., 2000 copies.
Symposium on Testing Building Construction (STP 312), 89 pp., 2000 copies.
ASTM Manual on Fitting Straight Lines (STP 313), 34 pp., 2000 copies.
Reactor Structural Materials—Engineering Properties as Affected by Nuclear Reactor Service, by W. K. Anderson, C. J. Beck, A. R. Kephart, and J. S. Theilacker (STP 314), 100 pp., 2500 copies.
1962 Engine Test Sequences for Evaluating Oils for API Service Ms (STP 315), 26 pp., 2000 copies.
Bibliography and Abstracts on Primary Soil Deposition 1931–1961 (STP 316), 21 pp., 1200 copies.
Advances in Electron Metallography and Electron-Probe Microanalysis (STP 317), 213 pp., 2000 copies.
Symposium on Electroforming—Applications, Uses and Properties of Electroformed Metals (STP 318), 210 pp., 2500 copies.
Symposium on Soldering (STP 319), 98 pp., 2000 copies.
Symposium on Turbine Oils (STP 321), 118 pp., 2000 copies.
Microstructure of Bronze Sinterings, by E. W. Lowe and H. R. Biehl (STP 323), 8 pp., 2000 copies.
Symposium on Stress-Strain-Time-Temperature Relationships in Materials (STP 325), 134 pp., 2500 copies.
Symposium on Skid Resistance (STP 326), 120 pp., 2000 copies.
Space Radiation Effects on Materials (STP 330), 69 pp., 3000 copies.
Molecular Formula List of Compounds, Names and References to Published Infrared Spectra (STP 331), 316 pp., 2000 copies.
ASTM Manual for Conducting an Interlaboratory Study of Test Methods (STP 335), 47 pp., 2000 copies.

Special Publications in Prospect:

Five Year Bibliography on Fatigue 1950–1954 (STP 9-AA).
Supplement to Bibliography and Abstracts on Electrical Contacts (STP 56-Q).
Tentative Guide for Fatigue Testing and the Statistical Analysis of Fatigue (STP 91-B).
Physical Constants of Hydrocarbons Boiling Below 350 F (STP 109).
Symposium on Masonry Testing (STP 320).
Symposium on Field Tests and Measurements for Soils and Foundation Engineering (STP 322).
Symposium on Simulated Service Testing of Packaging (STP 324).
Symposium on Standards for Filament-Wound Reinforced Plastics (STP 327).
Symposium on Fundamental Viscosity of Bituminous Materials (STP 328).
Coden for Periodical Titles (STP 329).
Manual for Hydrocarbon Analysis (STP 332).
Directory of Testing Laboratories—Commercial, Institutional (STP 333).
Symposium on Lubricants for Automotive Equipment (STP 334).
Symposium on Dynamic Behavior of Materials (STP 336).
Symposium on Water and Industrial Waste Water (STP 337).
* Symposium on Fatigue of Aircraft Structures (STP 338).
Symposium on Advances in Techniques in Electron Metallography (STP 339).

* Symposium on Properties of Surfaces (STP 340).
* Symposium on Radiation Effects on Metals and Neutron Dosimetry (STP 341).
* Symposium on Cleaning and Materials Processing for Electronics and Space Apparatus (STP 342).
* Symposium on Fire Test Methods (STP 344).
Gas Chromatographic Data (STP 345).
Symposium on Electrical Insulating Gases (STP number not yet assigned).
1963 Annual Meeting Symposiums.

Respectfully submitted on behalf of the committee,

R. E. HESS,
Chairman.

Jane B. Wheeler
Secretary.

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* In addition to the above there are other symposia resulting from the Fourth Pacific Area National Meeting.

APPENDIX V

REPORT OF THE ADMINISTRATIVE COMMITTEE ON DISTRICT ACTIVITIES

During the year, the Administrative Committee on District Activities held three meetings: at the Annual Meeting in New York City, June 26, 1962; at the Pacific Area National Meeting in Los Angeles, Oct. 1, 1962; and at National Headquarters in Philadelphia, Jan. 16, 1963.

At the meeting in June, the following joined the committee: Francisco Chairez, L. F. Herron, E. N. Klemgard, Bryant Mather, G. D. Patterson, Jr., and B. E. Tiffany. L. A. O'Leary was reappointed chairman of the committee.

A Manual for Student Award Presentation was compiled and distributed to the District Councils. Each district was also presented with an ASTM banner to display at its meetings. In addition, a brochure describing the benefits of Student Membership Prize Awards was prepared and is now distributed to each awardee.

The committee sponsored three open sessions during the year. At the Annual Meeting, a four-man panel discussed public relations and a three-man panel discussed communications between districts, national headquarters, and the members-at-large. At the Pacific Area National Meeting, a three-man panel discussed "How to Hold a Successful Meeting."

Four new districts were formed during the year: Hawaii, Oklahoma, North Texas, and Mexico. The latter was the first district formed entirely outside the United States. In addition, all of Canada not previously affiliated with ASTM districts was associated with neighboring districts in the United States. The formation of a new district in the Mississippi Delta area is under consideration.

During the year, 22 of the 24 districts held at least one meeting for a total of 29 meetings. Of these, 12 were jointly sponsored or held in cooperation with other technical groups.

The Student Award Program continues to expand, and some 800 awards were granted through 15 active district programs. Two more districts are seriously considering activating a Student Award Program.

The Chicago District continued its Speakers' Bureau, which is serving as the model for other districts contemplating similar activity.

The Northern California District Council arranged a one-day program centered around ASTM activities for members of the Asian Productivity Organization on Feb. 19, 1963, in San Francisco. This group toured the United States for two weeks on a study of quality control and standardization under the sponsorship of the Agency for International Development. Representatives came from Japan, Republic of China, India, Korea, Nepal, Pakistan, Philippines, and Thailand. This is a typical activity in which districts can promote the over-all interests of the Society.

The Administrative Committee on District Activities has undertaken an extensive study of district activities and objectives. There will be an open session at the 1963 Annual Meeting in which officers, district council members, and all others interested in district activities will be invited to participate. As a result of the open session and the survey, the committee expects to make positive suggestions to the Staff and to the Board of Directors to strengthen the District Program in a definitive report.

A complete summary of all district meetings and other activities may be found in the Annual Report of the Board of Directors under District and Related Activities.[1]

Respectfully submitted on behalf of the committee,

L. A. O'LEARY,
Chairman.

A. L. BATIK,
Secretary.

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[1] See p. 63.

APPENDIX VI

REPORT OF THE ADMINISTRATIVE COMMITTEE ON SIMULATED SERVICE TESTING

Two meetings of the Administrative Committee on Simulated Service Testing have been held during the period covered by this report: on June 25, 1962, in New York City, and on Feb. 4, 1963, in Montreal, Canada.

The Symposium on Packaging (*ASTM STP No. 324*) sponsored by this committee at the 1962 Annual Meeting has been issued. This publication contains a paper on the basic mechanics of packaging and four papers covering practical aspects in various fields of application.

The 1961 Symposium on Erosion and Cavitation (*ASTM STP No. 307*) led to considerable interest in further activity, particularly on the part of participants from the Naval Laboratories. At the recommendation of the Division of Materials Sciences, a meeting may be held to determine the feasibility of a research group for the study of erosion.

A Symposium on Materials Problems in the Design of a Supersonic Transport Aircraft was developed for the 1963 Annual Meeting. The following program was organized by H. F. Hardrath (NASA), a member of the Administrative Committee:

The Supersonic Transport Project: Operating Environment and Materials Problems, by F. R. Kostock, North American Aviation Inc.
Materials for Wings and Fuselage of a Supersonic Transport, by R. H. Raring, National Aeronautics and Space Administration
Sheet Material for Supersonic Transport, by H. Hightchew, The Boeing Airplane Co.
Fuels and Lubricants for the Supersonic Transport, by M. Dunnam, Wright-Patterson Air Force Base

A. G. Norem of Aerospace Corp. has been added to the committee to represent the space field.

E. F. Seaman was appointed as liaison representative between the committee and the Institute of Environmental Sciences, a society in which he has been active.

Topics under discussion are building construction, wear and galling, biomechanics (surgical implants, seat belts), effects at high pressure, and effects under extreme vacuum conditions.

Respectfully submitted on behalf of the committee,

R. E. Peterson,
Chairman.

S. Etris,
Secretary.

APPENDIX VII

REPORT OF THE ADMINISTRATIVE COMMITTEE ON EDUCATION IN MATERIALS

The Administrative Committee on Education in Materials met December 28, 1962. It discussed at considerable length the many problems facing educators, industry, and government agencies with regard to research related to new materials, and the problems to be faced in developing standards rapidly enough and stringent enough to meet our present-day needs.

The committee reviewed its scope and the explanatory note as set forth in the report of the committee to the Board of Directors, October 31, 1958.

> *Scope.*—This committee is to advise the Board of Directors on what the Society should be doing and what it appropriately can do in the field of engineering and science education in materials, and to implement the program.
>
> *Explanatory Note.*—This broad scope might encompass promoting an interest in engineering education and science; developing studies on important subjects to be included within existing curricula; recognizing achievements in this field both among faculty members and engineering and science students; and in general sponsoring activities that will stimulate interest in ASTM and its committee assignments which will afford the Society a steadily growing reservoir of personnel from which it can derive members and committee members to carry on its current and new work.

The Scope was considered appropriate as written except that the last phrase "...and to implement the program" placed the activities required beyond the manpower of a small committee. It was believed that for this reason, previous efforts to get work under way were unsuccessful.

An attempt to develop coordination between the Committee on Education in Materials and interested technical committees of the Society was not fruitful. Officers of five technical committees were approached for advice and help in furthering the work of the committee through the preparation of technical articles specific to their interests, for suggestions regarding the teaching of the science of materials, and for suggestions on the preparation of instructional aids for teaching or reference. The responses indicated that education in materials is considered an important subject, but no suggestions were offered for developing a program of assistance.

The committee agreed that it would be very difficult for any technical or professional society, such as ASTM, to influence educational programs at a college or university. Educators are notably independent; and if they are doing the type of work they are expected to do in research, they should be ahead of the Society itself with the latest information on research.

The committee questioned whether or not ASTM, without coordination with the American Society for Engineering Education, should attempt to develop curricula for training in engineering materials. This subject would seem to fall within the scope of the activities of that organization.

The Division of Materials Sciences, which was organized after the Committee on Education in Materials, is now working on subjects parallel to those which have fallen within the scope of the committee. Since the DMS has a much larger group with broader interests and more resources, and is in a better position to be of assistance in engineering education than the present committee, the Administrative Committee on Education in Materials recommended that it should be discharged and its responsibilities transferred to the Division of Materials Sciences.

The Division of Materials Sciences' Executive Committee has agreed with this recommendation and the committee has proposed to the Board of Directors that it be discharged and that its responsibilities be transferred to the Division of Materials Sciences.

Respectfully submitted on behalf of the committee,

M. J. SINNOTT,
Chairman.

FRED F. VAN ATTA,
Secretary.

APPENDIX VIII

REPORT OF THE ADMINISTRATIVE COMMITTEE ON INTERSOCIETY RELATIONS

As an outgrowth of the recommendations of the Long-Range Planning Committee, the Board of Directors accepted the recommendation to establish an Administrative Committee on Intersociety Relations having the following scope:

(*a*) To maintain a continuing review of the Society's contacts and relations with other societies.

(*b*) To recommend policies and procedures for improving and maintaining effective intersociety relations between ASTM and other organizations.

(*c*) To develop a specific program to broaden our relations with other societies and organizations in areas that will further promote the accomplishment of the objectives of ASTM.

The details of appointment and composition of the committee were resolved by the Board at its meeting in May, 1962, and the new administrative committee came into being, replacing the existing Board Committee on Inter-Society Relations.

The first meeting of the committee was held on Sept. 14, 1962, at ASTM temporary headquarters, 3001 Walnut St., Philadelphia, Pa. Among the actions taken at this first meeting was a review of basic ASTM policy on intersociety relations as reflected in a statement issued to ASTM representatives to other organizations. This statement was reviewed by the committee in juxtaposition with statements by other societies such as the American Chemical Society. The committee reaffirmed the ASTM statement as basic policy but suggested some rephrasing to provide for broader use.

The committee reviewed relations with the American Standards Assn., American Society for Metals, Instrument Society of America, Engineers Joint Council, Steel Structures Painting Council as well as several minor problems involving chemical abstracts and absorption spectral data. The next meeting of the committee will be held in Atlantic City, N.J., during the 1963 Annual Meeting.

In addition, the following items are pertinent to the first annual report of the committee.

ASTM-ASM Liaison Committee:

Although not formally organized within the framework of the Committee on Intersociety Relations, the ASTM-ASM Liaison Committee deals with matters of direct interest to the Intersociety Committee and will report herewith through that committee.

The ASTM-ASM Liaison Committee has had one meeting during 1962–1963 at which all representatives from both Societies were present. The purpose of this first meeting was entirely exploratory, and no precise agenda of action was pursued. The Liaison Committee, however, established a cordial agreement on the nature of the problems jointly confronting ASTM and ASM. It was agreed that these problems would be solved only by continued attention and action which will probably require several years of negotiations. A second meeting of the Committee was planned for early spring 1963, but due to heavy prior commitments on the part of various members, a second meeting has not yet been convened. It is currently the hope that this second meeting will occur shortly, at which time a series of expressed actions may be undertaken.

College Placement Offices:

At a meeting of the Engineering Manpower Commission, Engineers Joint Council, held March 21, 1963, at which A. Allan Bates was present as a delegate from ASTM, there was a long discussion about efforts of the U. S. Employment Service to develop employment placement offices on college campuses.

Members of the various government agencies, including USES, were present, together with representatives from many private industries and associations. The argument was long and unrestrained with the final outcome as set forth in the following paragraph:

> It is the position of the Engineering Manpower Commission that the most effective approach to the placement of graduating engineers is accomplished by direct negotiation between the engineering candidate and employer within the framework of facilities provided by the college or university. The Commission believes the interposing of a third party between the employer and candidate is neither desirable nor necessary and detracts from the objective of establishing early the professional relationships. Accordingly, the Commission takes the position that recent efforts of the USES to expand its present activities to displace the normal college placement functions are not in the best interests of the students, employers, and professional societies.

This quoted paragraph was adopted by majority vote. Since ASTM was represented at the meeting by its appointed member to the Manpower Commission, the Society is, to a degree, committed to the sense of the resolution.

Foreign Chapters of Technical Societies:

The Committee on International Relations of the Engineers Joint Council has been considering the implications of growth in foreign memberships of American societies. This has involved, among other things, the establishment of foreign chapters of various American technical societies.

The American Society of Civil Engineers is considering adoption of a policy which will discourage the authorization and formation of additional ASCE sections outside the United States. The EJC is studying and may recommend a similar policy for general adoption by American engineering societies.

ASTM has recently established a district in Mexico. Some consideration has been given to the formation of districts in other Latin American countries. Furthermore, ASTM has many foreign members. The wide adoption by American engineering societies of an EJC recommendation relating to foreign activities might materially affect ASTM policies. The ASTM Committee on Intersociety Relations should watch this development closely and take such action, or make such proposals, as may be necessary for the protection of ASTM interests.

Respectfully submitted on behalf of the committee,

A. ALLAN BATES,
Chairman.

REPORT OF COMMITTEE A-1
ON
STEEL*

During the past year, Committee A-1 has been very active in reviewing the specifications under its jurisdiction with the aim of producing consistency and clarity. The specifications are being studied particularly for the removal of all open-end agreements and statements.

Several specifications for special-purpose wire and a general requirement specification for steel wire are well along in the development stage. Also several new grades are being considered for addition to existing specifications.

Work continues in two subcommittees on specifications for quenched-and-tempered alloy steels for structural steel and pressure vessel applications. Users have also requested a specification for forgings made from these steels. Another specification for 9 per cent nickel steel forgings is to be developed.

Other specifications either to be drafted or well under way are for: low-alloy steel castings suitable for pressure service, high hardenability bearing steels, special quality ball and roller bearing steels, and high-strength cold-drawn bars. Also to be written is a recommended practice for the ultrasonic examination of nonmagnetic retaining rings for generators.

Subcommittee XXIX on Tool Steel held an organization meeting on Jan. 22, 1963. A task group was appointed to develop a policy for the subcommittee and to recommend compositions and types of tool steels to be included in the initial specifications. This group is to meet in April and it expects to have a report ready for the Annual Meeting in June on the direction that Subcommittee XXIX will take. Other producers and consumers will be invited to participate in the subcommittee activity.

For several years a task group of Committee A-1 officers has been working on a revision of the committee's bylaws to bring them up to date with ASTM procedures and particularly to clarify provisions for standardization procedures. The proposed bylaws were approved by Committee A-1 letter ballot dated May 16, 1962, with one negative vote objecting to elimination of references to tentatives until the Society has approved a procedure for revision of standards between Annual Meetings. The Advisory Committee voted to retain as tentative all tentative A-1 specifications and methods until such a procedure is finalized.

Committee A-1 approved the submittal of all A-1 specifications and methods to the American Standards Assn. for adoption as American Standards. This is handled individually as each specification is revised and the revision is balloted by the main committee.

The Advisory Committee authorized the chairman of Subcommittee XIII on Methods of Testing to work with Committee E-4 on Metallography in revising

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

Method E 112 so that proper note would be taken of different procedures such as outlined in Federal Test Standard 151a.

Committee A-1 is interested in including metric equivalents in its specifications, but will await further advice from the ad hoc committee appointed by the Board of Directors to make recommendations on equivalents to be used before making any extensive revisions.

U. S. A. National Committee for ISO/TC 17 on Steel.—The U. S. A. National Committee for ISO/TC 17 met on June 26, 1962, in New York, N. Y., and on Jan. 22, 1963, in Detroit, Mich. During the year the U. S. A. National Committee arranged for representation as follows at Working Group meetings:

Working Group 1 on Methods of Mechanical Testing, May 7–9, 1963, Glasgow, Scotland, A. G. Cook.

Working Group 3 on Structural Steels, March 4–6, 1963, Paris, France, J. R. LeCron.

Working Group 4 on Heat Treated and Alloyed Steel, September 10–13, 1962, Dusseldorf, Germany, A. W. MacLaren.

Working Group 7 on Methods of Test Other Than Mechanical and Chemical Analysis, January 10–16, 1963, Paris, France, W. H. Mayo.

Working Group 8 on Redesign of Hot-Rolled Steel Section, January 22–24, 1963, New Delhi, India, F. E. Fahy.

Working Group 9 on Tinplate, March 12–14, 1963, London, England, F. F. Aloi. There is no present activity in Committee A-1 or any other ASTM technical committee on this subject.

The personnel in all above cases was not drawn from the personnel of Committee A-1.

The U. S. A. National Committee has also during the past year arranged for letter ballot consideration of the appropriate groups within ASTM on the following Draft ISO Recommendations:

I.S.O. No.	*Title*
516	General Principles for Fatigue Testing
517	Interrupted Creep Testing of Steel at Elevated Temperatures (Temperature, Not Load, Interrupted)
518	Ring Expanding Test on Steel Tubes
519	Tensile Testing of Steel Tubes
520	Calibration of Pendulum Impact Testing Machines
521	Calibration of Elastic Proving Devices
522	Calibration of Rockwell B and C Scale Hardness Testing Machines
526	General Technical Delivery Requirements for Steel
527	Selection and Preparation of Samples and Test Pieces

As a result of these letter ballots, the U. S. A. has cast affirmative ballots on Draft ISO Recommendations 518, 519, 521, 522, and 526, and negative ballots on Draft ISO Recommendations 516, 517, and 520.

U. S. A. National Committee of the International Deep Drawing Research Group.—A meeting of the group was held on Sept. 27, 1962, in Detroit, Mich. U. S. A. representatives attended the Working Group meetings of the IDDRG on May 24, 1962, in Düsseldorf, Germany. A cooperative program on an international basis has been arranged in the Materials Group measuring deep drawing characteristics. Samples of one steel have been tested in several countries by the method preferred by the individual laboratory. The next step is for the chairman of the Materials Group to suggest one method for measuring each value for a cooperative program.

There will be a meeting of the Working Groups of the IDDRG in Stockholm, Sweden, on June 17 and 18, 1963.

Meetings.—Committee A-1 met on June 27, 1962, in conjunction with the 1962 Annual Meeting in New York, N. Y. Twenty subcommittees, sections, and task groups met on June 25 and 26 to review the past year's activities, to consider the results of recent ballots, and to organize work for the coming year.

A joint meeting of the advisory groups

of Committees A-1 and A-10 was held Jan. 10, 1963, in New York City to discuss the work and scheduling of meetings of the two committees.

On Jan. 23, 1963, Committee A-1 met in Detroit, Michigan. On Jan. 20, 21, and 22, twenty-five subcommittee, section, and task group meetings were held. This included the first meeting of the newly formed Subcommittee XXIX on Tool Steel.

Personnel.—The committee consists of 355 voting members, including 160 producers, 141 consumers, and 54 general interest members.

The following special appointments were made: I. A. Rohrig as ASTM representative on ASA Sectional Committee B36, and J. E. Lattan as ASTM representative on ASA Sectional Committee B16.

Joseph Field of Bethlehem Steel Co. was appointed as chairman of Subcommittee XV on Bar Steels to replace H. N. Meyer who resigned because of change of duties in his company. A. S. Jameson of International Harvester Co. was designated chairman of the new Subcommittee XXIX on Tool Steel. F. A. Webber, Colorado Fuel and Iron Corp., is replacing C. L. Kent as Chairman of Subcommittee III on Steel and Wire.

Four members of the committee were lost in 1962 by death: W. F. Collins, Past-Chairman of A-1; Ernest F. Lundeen, Honorary Member of A-1 (longtime chairman of Subcommittee XIX on Sheet Steel); H. B. Oatley, Honorary Member of A-1 and the Society and Past-Chairman of A-1; and Fred B. Riggan, member of A-1 since 1955.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

The Administrative Committee on Standards, acting for the Society on the dates shown, accepted the following recommendations of Committee A-1:

Accepted on July, 23 1962:

Revision and Reversion to Tentative of Standard Specifications for (Jointly with Committee A-10):

Seamless and Welded Ferritic Stainless Steel Tubing for General Service (A 268 - 62),
Seamless and Welded Austenitic Stainless Steel Tubing for General Service (A 269 - 62),
Seamless and Welded Austenitic Stainless Steel Sanitary Tubing (A 270 - 62), and
Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 - 61).

Accepted on July 27, 1962:

New Tentative Specifications for:

Vacuum Treated Steel Forgings for Generator Rotors (A 469 - 62 T),
Vacuum Treated Carbon and Alloy Steel Forgings for Turbine Rotors and Shafts (A 470 - 62 T), and
Vacuum Treated Alloy Steel Forgings for Turbine Rotor Disks and Wheels (A 471 - 62 T),

New Tentative Method for:

Heat Stability Test on Steam Turbine Shafts and Rotor Forgings (A 472 - 62 T).

Revision of Tentative Specifications for:

Seamless Carbon Steel Boiler Tubes (A 83 - 62 T),
Seamless Carbon-Steel Pipe for High-Temperature Service (A 106 - 62 T),
Seamless Low-Carbon and Carbon-Molybdenum Steel Still Tubes for Refinery Service (A 161 - 61 T),
Electric-Resistance-Welded Carbon Steel Boiler Tubes (A 178 - 62 T),
Seamless Cold-Drawn Low-Carbon Steel Heat-Exchanger and Condenser Tubes (A 179 - 62 T),
Seamless Carbon Steel Boiler Tubes for High-Pressure Service (A 192 - 62 T),
Alloy-Steel Bolting Materials for High-Temperature Service (A 193 - 61 T),
Carbon and Alloy Steel Nuts for Bolts for High-Pressure and High-Temperature Service (A 194 - 59 T),
Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes (A 199 - 62 T),

Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service (A 200 - 61 T),
Seamless Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 209 - 61 T),
Seamless Medium-Carbon Steel Boiler and Superheater Tubes (A 210 - 62 T),
Seamless Ferritic and Austenitic Alloy-Steel Boiler, Superheater, and Heat Exchanger Tubes (A 213 - 62 T),
Electric-Resistance-Welded Carbon Steel Heat-Exchanger and Condenser Tubes (A 214 - 62 T),
Electric-Resistance-Welded Carbon Steel Boiler and Superheater Tubes for High-Pressure Service (A 226 - 61 T),
Electric - Resistance - Welded Carbon - Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 250 - 61 T),
Seamless and Welded Ferritic Stainless Steel Tubing for General Service (A 268 - 60 T),
Carbon and Alloy Steel Forgings for Turbine Rotors and Shafts (A 293 - 60 T),
Alloy-Steel Bolting Materials for Low-Temperature Service (A 320 - 59 T),
Seamless and Welded Steel Pipe for Low-Temperature Service (A 333 - 62 T),
Seamless and Welded Carbon and Alloy-Steel Tubes for Low-Temperature Service (A 334 - 62 T),
Seamless Ferritic Alloy Steel Pipe for High-Temperature Service (A 335 - 62 T),
Seamless Ferritic Alloy-Steel Pipe Specially Heat-Treated for High-Temperature Service (A 405 - 62 T), and
Seamless and Electric Welded Low Alloy Steel Tubes (A 423 - 62 T).

Accepted on September 11, 1962:

Revision of Tentative Specifications for:

Seamless Carbon Steel Boiler Tubes (A 83 - 62 T),
Seamless Low-Carbon and Carbon-Molybdenum Steel Still Tubes for Refinery Service (A 161 - 61 T),
Electric-Resistance-Welded Carbon Steel Boiler Tubes (A 178 - 62 T),
Seamless Cold-Drawn Low-Carbon Steel Heat-Exchanger and Condenser Tubes (A 179 - 62 T),
Seamless Carbon Steel Boiler Tubes for High-Pressure Service (A 192 - 62 T),
Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes (A 199 - 62 T),
Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service (A 200 - 61 T),
Seamless Carbon - Molybdenum Alloy - Steel Boiler and Superheater Tubes (A 209 - 61 T),
Seamless Ferritic and Austenitic Alloy-Steel Boiler, Superheater, and Heat-Exchanger Tubes (A 213 - 62 T),
Electric-Resistance-Welded Carbon Steel Heat-Exchanger and Condenser Tubes (A 214 - 62 T),
Electric-Resistance-Welded Carbon Steel Boiler and Superheater Tubes for High-Pressure Service (A 226 - 61 T),
Welded Austenitic Steel Boiler, Superheater, Heat-Exchanger, and Condenser Tubes (A 249 - 62 T),
Electric - Resistance - Welded Carbon - Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 250 - 61 T),
Copper Brazed Steel Tubing (A 254 - 61 T),
Seamless and Welded Ferritic Stainless Steel Tubing for General Service (A 268 - 62 T),
Seamless and Welded Austenitic Stainless Steel Tubing for General Service (A 269 - 62 T),
Seamless and Welded Austenitic Stainless Steel Sanitary Tubing (A 270 - 62 T),
Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 - 62 T),
Seamless and Welded Carbon and Alloy-Steel Tubes for Low-Temperature Service (A 334 - 62 T),
Quenched and Tempered Alloy Steel Bolts and Studs with Suitable Nuts (A 354 - 58 T),
Seamless and Electric Welded Low Alloy Steel Tubes (A 423 - 62 T), and
General Requirements for Carbon, Ferritic Alloy, and Austenitic Alloy Steel Tubes (A 450 - 62 T).

Accepted on October 10, 1962:

Revision of Tentative Specification for:

Structural Steel (A 36 - 61 T).

Accepted April 11, 1963:

Revision of Tentative Specification for:

High-Strength Structural Steel (A 440 - 59 T), and
High-Strength Low Alloy Structural Manganese Vanadium Steel (A 441 - 60 T).

Revision and Reversion to Tentative of Standard Specification for:

High Strentgh Low Alloy Structural Steel (A 242 - 60).

Accepted April 00, 1963:

New Tentative Specification for:

Deformed Rail Steel Bars for Concrete Reinforcement with 60,000 psi Minimum Yield Strength (A 61 - 63 T).

Revision of Tentative Specification for:

Rail Steel Bars for Concrete Reinforcement (A 16 - 62 T).

The new and revised specifications appear in the 1962 Supplement to the Book of ASTM Standards, Part 1, with the exception of those accepted in April, 1963, which are available as separate reprints.

RECOMMENDATIONS AFFECTING STANDARDS

Committee A-1 requests approval by the Society at the Annual Meeting of the recommendations given in detail in the Appendix.[1] The results of the committee ballot on the recommendations will be reported at the Annual Meeting.

This report has been submitted to letter ballot of the committee, which consists of 355 members; 231 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. J. KANTER,
Chairman.

H. R. BOATMAN,
Secretary.

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[1] See p. 115.

APPENDIX

PROPOSED RECOMMENDATIONS AFFECTING STANDARDS ON STEEL

Committee A-1 requests approval by the Society at the Annual Meeting of the recommendations given in detail in this Appendix, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1]

The specifications under revision appear in their present form in the 1961 Book of ASTM Standards, Part 1, or its 1962 Supplement, unless otherwise noted.

New Standard

(requires nine-tenths affirmative vote)

Standard Specification for High Hardenability Bearing Steels, as appended hereto.[2]

New Tentatives

Tentative Specifications for:

Eyebolts,[2]
Castings for Highway Bridges,[3]
Low-Alloy Steel Castings Suitable for Pressure Service,[3] and
Quenched and Tempered Steel Bolts and Studs.[2]

Tentative Recommended Practice for:

Qualification of Procedures and Personnel for the Welding of Steel Castings.[3]

Revisions of Tentatives

Tentative Specification for General Requirements for Delivery of Rolled Steel Plates of Flange and Firebox Qualities (A 20 – 62 T):

Section 9.—Delete reference to butt straps in Paragraph (*a*). Delete Paragraphs (*d*), (*e*), (*f*), and (*g*).

Tentative Specification for Welded and Seamless Steel Pipe (A 53 – 62 T):

Section 7.—Replace Paragraphs (*c*) and (*d*) covering the flattening test for electric-resistance welded pipe by Section 7(*a*) and (*b*) of Specification A 135.

Section 15(*a*).—Delete Paragraph (*3*).

Table V.—For butt-welded pipe, add the following test pressures:

Nominal Size, in.	Wall Thickness, in.	Test Pressure, psi
3	0.188	1000
4	0.156	1000
3½	0.188	1200
4	0.188	1200
4	0.219	1200

Table VI.—For butt-welded pipe revise test pressures from "800" psi to "1000" psi for the following sizes: 1¼ by 0.140 in. wall, 1½ by 0.145 in. wall, 2½ by 0.203 in. wall, and 3 by 0.216 in. wall.

Appendix.—Delete Paragraph 1A defining bell-welded pipe since it is no longer manufactured, and renumber subsequent paragraphs accordingly.

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[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[2] This new or revised specification appears in the *1964 Book of ASTM Standards*, Part 4.

[3] This new or revised specification appears in the *1964 Book of ASTM Standards*, Part 2.

Tentative Specification for Seamless Carbon-Steel Pipe for High-Temperature Service (A 106 – 62 T):

Section 8.—Designate present Section 8 as 8(*a*), and add a new Paragraph (*b*) to read as follows:

(*b*) For pipe whose diameter exceeds 25 in. and whose diameter to wall thickness ratio is 7.0 or less, bend test specimens shall be bent at room temperature through 180 deg without cracking on the outside of the bent portion. The inside diameter of the bend shall be 1 in. This test shall be in lieu of the flattening test.

Section 10.—Change definitions of "*t*" and "*D*" to read as follows:

t = specified wall thickness, in inches, and
D = specified outside diameter, in inches.

Section 11.—Revise Paragraph (*c*) and add a new Paragraph (*d*) to read as follows:

(*c*) Test specimens for the bend test specified in Section 8 and for the flattening tests shall consist of sections cut from a pipe. Specimens for flattening tests shall be smooth on the ends and free from burrs, except when made on crop ends.

(*d*) Test specimens for the bend test specified in Section 8(*b*) shall be cut from one end of the pipe and, unless otherwise specified, shall be taken in a transverse direction. One test specimen shall be taken as close to the outer surface as possible and another from as close to the inner surface as possible. The specimens shall be either $\frac{1}{2}$ by $\frac{1}{2}$ in. in section or 1 by $\frac{1}{2}$ in. in section with the corners rounded to a radius not over $\frac{1}{16}$ in. and need not exceed 6 in. in length. The side of the samples placed in tension during the bend shall be the side closest to the inner and outer surface of the pipe respectively.

Designate present Paragraph (*d*) as Paragraph (*e*).

Section 12(b).—Add the following sentence at the end of the paragraph: "The bend tests specified in Section 8(*b*) shall be made on one end of each pipe."

Tentative Specification for Black and Hot-Dipped Zinc-Coated (Galvanized) Welded and Seamless Steel Pipe for Ordinary Uses (A 120 – 62 T):

Table II.—In Footnote *e*, change definitions of "*t*" and "*D*" as indicated above for Section 10 of Specification A 106.

Tentative Specification for Electric-Resistance-Welded Steel Pipe (A 135 – 62 T):

Section 8(a).—Change definitions of "*t*" and "*D*" as indicated above for Section 10 of Specification A 106.

Tentative Specification for Electric-Fusion-Welded Steel Pipe for High-Temperature Service (A 155 – 62 T):

Section 12(a).—Change definitions of "*t*" and "*D*" to read as follows:

t = specified wall thickness, in inches, and
D = specified or calculated (from the specified inside diameter and wall thickness) outside diameter, in inches.

Tentative Specification for Carbon-Steel Castings Suitable for Fusion Welding for High-Temperature Service (A 216 – 60 T):

This specification has been extensively revised and is appended hereto in its proposed revised form.[3]

Tentative Specification for Carbon Steel Forgings for General Industrial Use (A 235 – 62 T):

Section 1(b).—Change Class F1 to read "normalized and tempered or double normalized and tempered."

Section 3.—Change Paragraph (*a*) to read: "The steel shall be made by any of the following processes: open-hearth, electric furnace or basic oxygen."

In Paragraph (*d*)(*2*), change Class F1 to read "normalized and tempered or double normalized and tempered."

Change Paragraph (*d*)(*3*) to read "Unless otherwise agreed upon by the manufacturer and the purchaser, no solid forging over 12 in. in diameter, and no bored forging over 10 in. in wall thickness shall be liquid quenched. For bored forgings with wall thickness over 5 in., the proportions of the forging must be such as to permit effective quenching."

Table II.—Change to read as shown in the accompanying Table I.[4]

Tentative Specification for Alloy Steel Forgings for General Industrial Use (A 237 - 62 T):

Section 1.—In Paragraph (*b*) after Classes B and C, insert a new class to read: "*Class C1*, normalized and tempered or double normalized and tempered."

Change Paragraph (*c*) to begin "Supplementary requirements (Paragraphs S1 to S8 . . .)"

Section 3.—Change Paragraph (*a*) to read "The steel shall be made by any of the following processes: open-hearth, electric furnace, or basic oxygen."

Change Paragraph (*d*)(*2*) to read as follows:

> 3.(*d*) (*2*) *Size Restrictions.*—Unless otherwise agreed upon by the manufacturer and the purchaser, for classes D and E, no solid forging over 20 in. in diameter, and no bored forging over 10 in. in wall thickness shall be liquid quenched; for classes F, G, and H no solid forgings over 10 in. in diameter, and no bored forgings over 5 in. in wall thickness shall be liquid quenched. For bored forgings, the proportions of the forging must be such as to permit effective quenching.

Section 7(b).—Change the first sentence to read: "Unless otherwise specified, any rejection based on tests made in accordance with the Section on Check Analysis shall be reported to the manufacturer within sixty days from the receipt of samples by the purchaser."

New Supplementary Requirements.—Add new Paragraphs S6 and S7 to read as follows, and renumber present S6 as S8:

> S6.—*Ultrasonic Test.*—When ultrasonic testing of forgings is required reference shall be made to the latest issue of Ultrasonic Testing and Inspection of Heavy Steel Forgings (ASTM Designation: A 388).
>
> S7.—*Vacuum Treatment.*—Vacuum treated steel shall be supplied when specified. The method of vacuum treatment shall be a matter of agreement between the manufacturer and the purchaser.

Table I.—Change to read as shown in the accompanying Table II, retaining the present footnotes.[4]

Tentative Specification for Carbon Steel Seamless Drum Forgings (A 266 - 62 T):

This specification has been extensively revised and is appended hereto in its proposed revised form.[5]

Tentative Specification for Carbon Steel Blooms, Billets, and Slabs for Forgings (A 273 - 58 T):

Section 2.—Change to read "The steel shall be made by the open-hearth, basic oxygen, or electric furnace process."

Supplementary Requirement S1.—Change to read "The purchaser may specify that the steel shall be made in the basic open-hearth, acid open-hearth, electric furnace, basic oxygen, or shall be vacuum melted. When agreed upon by the manufacturer and the purchaser the steel shall be vacuum poured."

New Supplementary Requirement S11.—Add a new Supplementary Requirement S11 entitled "Ultrasonic Testing" to read as follows renumbering subsequent supplementary requirements accordingly: "Each semifinished bloom, billet, or slab shall be ultrasonically tested to determine its soundness in a manner agreed upon by the manufacturer and the purchaser."

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[4] This table is not included in this report but appears in the revised tentative; see *1964 Book of ASTM Standards*, Part 4.

[5] This revised specification appears in the *1964 Book of ASTM Standards*, Part 1.

Tentative Specification for Alloy Steel Blooms, Billets, and Slabs for Forgings (A 274 – 58 T):

Section 2.—Change as indicated above for Section 2 of Specification A 273.

Supplementary Requirements.—Change as indicated above for Supplementary Requirements S1 and S11 of Specification A 273.

New Supplementary Requirements.—Add as indicated above for Specification A 273.

Tentative Specification for Low-Carbon Steel Externally and Internally Threaded Standard Fasteners (A 307 – 61 T):

Section 7(a).—Change the first sentence to begin "The requirements of this specification shall be met in continuous mass production . . ."

Section 10.—Change reporting time from "five" to "thirty" days.

Tentative Specification for Seamless and Welded Austenitic Stainless Steel Pipe (A 312 – 62 T):

New Section 3.—Add a new Section 3 entitled "Basis of Purchase" to read as follows, renumbering the present section accordingly:

> 3. Orders for material under this specification shall include the following, as required, to describe the desired material adequately:
>
> (*1*) Quantity (feet or number of lengths),
> (*2*) Name of material (austenitic steel pipe),
> (*3*) Process (seamless or welded),
> (*4*) Grade (Table I),
> (*5*) Size (nominal size or outside diameter and schedule number or average wall thickness),
> (*6*) Length (specific or random, Section 17),
> (*7*) End finish (Section 18),
> (*8*) Optional requirements (check analysis, Section 8; supplementary requirements S1 to S5),
> (*9*) Test report required (Section 21(*b*)),
> (*10*) ASTM designation, and
> (*11*) Special requirements or exception to the specification.

Section 11(a).—Change definitions of "*t*" and "*D*" as indicated above for Section 10 of Specification A 106.

Tentative Specification for Alloy-Steel Bolting Materials for Low-Temperature Service (A 320 – 62 T):

Table I.—Change sulfur requirements of Grade 8F from "0.15 min" to "0.15 to 0.35" per cent.

Section 9(a).—Change the last sentence to read: "Impact tests are not required for grades B8D, B8F, B8M, and B8T for temperatures above −325 F; for Grades B8 and B8C above −425 F; or for bolts $\frac{1}{2}$ in. and smaller in diameter

Supplementary Requirement S1.—Delete the last sentence.

Tentative Specification for High-Strength Steel Bolts for Structural Steel Joints, Including Suitable Nuts and Plain Hardened Washers (A 325 – 61 T):

Section 4(g).—Change Rockwell hardness of quenched and tempered hardened washers from "C35 to C45" to read "C38 to C45."

Section 7(a).—Change the first sentence to begin "The requirements of this specification shall be met in continuous mass production . . ."

Section 10.—Change reporting time from "ten" days to "thirty" days.

Tentative Specifications for Seamless and Welded Steel Pipe for Low-Temperature Service (A 333 – 62 T):

New Section 3.—Add a new Section 3 entitled "Basis of Purchase" to read as follows, renumbering the present sections accordingly:

3. Orders for material under this specification shall include the following, as required, to describe the desired material adequately:

(1) Quantity (feet or number of lengths),

(*2*) Name of material (seamless or welded pipe),

(*3*) Grade (Table II),

(*4*) Size (nominal pipe size or outside diameter and schedule number or average wall thickness),

(*5*) Length (specific or random, Section 21),

(*6*) End finish (Section 22)

(*7*) Optional requirements (check analysis, Section 8; lower temperature for impact tests, Section 12(*a*),

(*8*) Test report required (Section 25(*b*),

(*9*) ASTM designation, and

(*10*) Special requirements or exceptions to specification.

Section 13(a).—Change definitions of "*t*" and "*D*" as indicated above for Section 10 of Specification A 106.

Tentative Specification for Seamless and Welded Carbon and Alloy-Steel Tubes for Low-Temperature Service (A 334 - 62 T):

New Section 2.—Add a new Section 2 entitled "Basis of Purchase" to read as follows, renumbering present sections accordingly:

2. Orders for material under this specification shall include the following, as required, to describe the desired material adequately:

(*1*) Quantity (feet or number of lengths),

(*2*) Name of material (seamless or welded tubes),

(*3*) Grade (Table I),

(*4*) Size (outside diameter and minimum wall thickness),

(*5*) Length (specific or random),

(*6*) Optional requirements (ladle or check analysis for welded tubes, Section 7; lower temperatures for impact tests, Section 12(*a*); hydrostatic or electric test, Section 14(*g*)),

(*7*) Test report required Section 22(*b*) of Specification A 450,

(*8*) ASTM designation, and

(*9*) Special requirements or exceptions to specification.

Tentative Specifications for Seamless Ferritic Alloy Steel Pipe for High-Temperature Service (A 335 - 62 T):

New Section 2.—Add a new Section 2 entitled "Basis of Purchase" to read as follows, renumbering the present sections accordingly:

2. Orders for material under this specification shall include the following, as required, to describe the desired material adequately:

(*1*) Quantity (feet or number of lengths),

(*2*) Name of material (seamless alloy steel pipe),

(*3*) Grade (Table I),

(*4*) Manufacture (hot-finished or cold-drawn),

(*5*) Size (nominal pipe size or outside diameter and schedule number or average wall thickness),

(*6*) Length (specific or random, Section 17),

(*7*) End finish (Section 18),

(*8*) Optional requirements (check analysis, Section 7; supplementary requirements, S1 to S7; hydrostatic test, Section 10, Note; weighing individual lengths, Section 16(*a*); repair by welding, Section 19(*d*)),

(*9*) Test report required (Section 21(*b*)),

(*10*) ASTM designation, and

(*11*) Special requirements or exceptions to the specification.

Section 9.—Designate present paragraph as (*a*), and add a new Paragraph (*b*) to read as follows:

(*b*) For pipe whose diameter exceeds 25 in. and whose diameter to wall thickness ratio is 7.0 or less, bend test specimens shall be bent at room temperature through 180 deg without cracking on the outside of the bent portion. The inside diameter of the bend shall be 1 in. This test shall be in lieu of the flattening test.

Section 10(a).—Change definitions of "*t*" and "*D*" as indicated above for Section 10 of Specification A 106.

Section 12.—Add new Paragraphs (*c*) and (*d*) to read as follows:

(*c*) Test specimens for the flattening tests shall consist of sections cut from a pipe, and shall be smooth on the ends and free from burrs except when made on crop ends.

(*d*) Test specimens for the bend test specified in Paragraph (*b*) shall be cut from one end of the pipe and, unless otherwise specified, shall be taken in a transverse direction. One test specimen shall be taken as close to the outer surface as possible and another from as close to the inner surface as possible. The specimens shall be either $\frac{1}{2}$ in. square in section or 1 by $\frac{1}{2}$ in. in section with the corners rounded to a radius not over $\frac{1}{16}$ in. and need not exceed 6 in. in length. The side of the samples placed in tension during the bend shall be the side closest to the inner and outer surface of the pipe respectively.

Designate present Paragraph (*d*) as (*e*), and delete present Paragraph (*c*).

Section 13(a).—Change to read as follows:

13 (*a*). For material heat treated in a batch-type furnace, tests shall be made as follows on 5 per cent of the pipe from each treated lot. For small lots at least one pipe shall be tested. Results of these shall be reported to the purchaser or his representative:

The transverse or longitudinal tension test specified in Section 8, and

The bend test specified in Section 9(*a*), and the flattening test specified in Section 9(*b*) for pipe over 25 in. in nominal diameter.

Tentative Specification for Ferritic and Austenitic Steel Castings for High Temperature Service (A 351 - 61 T):

Table I.—Add grade CN7M with the following chemical requirements:

Carbon, max, per cent	0.070
Manganese, max, per cent	1.50
Silicon, max, per cent	1.50
Sulfur, max, per cent	0.040
Phosphorus, max, per cent	0.040
Chromium, per cent	19.0 to 22.0
Nickel, per cent	27.5 to 30.5
Molybdenum, per cent	2.0 to 3.0
Columbium, per cent	3.0 to 4.0

Table II.—Add grade CN7M with with following mechanical properties:

Tensile strength, min, psi	62 500
Yield Strength, min, psi	25 000
Elongation in 2 in., min, per cent	35

Tentative Specification for Quenched-and-Tempered Alloy Steel Bolts and Studs with Suitable Nuts (A 354 - 62 T):

Section 10.—Change the reporting time from "five" to "thirty" days.

Tentative Specification for Electric-Fusion-Welded Austenitic Chromium-Nickel Alloy Steel Pipe for High-Temperature Service (A 358 - 62 T):

New Section 2.—Add a new Section 2 entitled "Basis of Purchase" to read as follows, renumbering the present sections accordingly:

2.—Orders for material under this specification shall include the following, as required, to describe the desired material adequately:

(*1*) Quantity (feet or number of lengths),

(*2*) Name of material (electric-fusion-welded pipe),

(*3*) Grade (Table I),

(*4*) Class (Section 1 (*c*)),

(*5*) Size (outside diameter and minimum wall thickness),

(*6*) Length (specific or random, Section 16),

(*7*) End finish (Section 17),

(*8*) Optional requirements (supplementary requirements S1 to S3; repair by welding, Section 18(*c*); sandblasting or pickling, Section 18(*d*)),

(*9*) Test report required,

(*10*) ASTM designation, and

(*11*) Special requirements or exceptions to specifications.

Section 12(a).—Change definitions of "*t*" and "*D*" as indicated above for Section 12(*a*) of Specifications A 155.

Tentative Specification for Carbon and Alloy Steel Forgings for Pressure Vessel Shells (A 372 - 62 T):

This specification has been extensively revised and is appended hereto in its proposed revised form.[2]

Tentative Specification for Seamless Austenitic Steel Pipe for High-Temperature Central-Station Service (A 376 - 62 T):

New Section 3.—Add a new Section 3 entitled "Basis of Purchase" to read as follows, renumbering the present sections accordingly:

3. Orders for material under this specification shall include the following, as required, to describe the desired material adequately:

(*1*) Quantity (feet or number of lengths),
(*2*) Name of material (seamless austenitic steel pipe),
(*3*) Grade (Table I),
(*4*) Size (nominal size, or outside diameter and schedule number or average wall thickness),
(*5*) Lengths (specific or random, Section 17),
(*6*) End finish (Section 18),
(*7*) Optional requirements (check analysis, Section 8; special hydrostatic tests, Section 11, Note 1; weighing individual lengths, Section 16; repair by welding, Section 19(*d*); retests on material, Section 21(*b*), supplementary requirements S1 to S7),
(*8*) Test report required (Section 21(*c*)),
(*9*) ASTM designation, and
(*10*) Special requirements or exceptions to specification.

Section 11(a).—Change definitions of "*t*" and "*D*" as indicated above for Section 10 of Specification A 106.

Tentative Specification for Galvanized Steel Transmission Tower Bolts and Nuts (A 394 - 61 T):

Section 11.—Change the reporting time from "five" to "thirty" days.

Tentative Specification for Seamless Ferritic Alloy-Steel Pipe Specially Heat-Treated for High-Temperature Service (A 405 - 62 T):

New Section 2.—Add a new Section 2 entitled "Basis of Purchase" to read as follows, renumbering the present sections accordingly:

2. Orders for material under this specification shall include the following, as required, to describe the desired material adequately:
(*1*) Quantity (feet or number of lengths),
(*2*) Name of material (alloy steel pipe),
(*3*) Grade (Table I),
(*4*) Size (nominal pipe size or outside diameter and schedule number or average wall thickness),
(*5*) Length (specific or random, Section 16),
(*6*) End finish (Section 17),
(*7*) Optional requirements (check analysis, Section 7; special hydrostatic test, Section 10, Note; repair by welding, Section 18(*d*); supplementary requirements S1 to S6),
(*8*) Test report required (Section 20(*b*)),
(*9*) ASTM designation, and
(*10*) Special requirements or exceptions to specification.

Section 10(a).—Change definitions of "*t*" and "*D*" as indicated above for Section 10 of Specification A 106.

Tentative Specification for Upholstery Spring Wire for Coiled Type Springs (A 407 - 57 T):

This specification has been extensively revised and is appended hereto in its proposed revised form.[6]

Tentative Specification for Welded Large Outside Diameter Light-Wall Austenitic Chromium-Nickel Alloy Steel Pipe for Corrosive or High-Temperature Service (A 409 - 60 T):

Section 9(a).—Change definitions of "*t*" and "*D*" as indicated above for Section 10 of Specification A 106.

Tentative Specification for Upholstery Spring Wire for Zig-Zag Type and Square Formed Type and No-Sag Type Spring Units (A 417 - 57 T):

This specification has been extensively revised and is appended hereto in its proposed revised form.[6]

Tentative Specification for Factory-Made Wrought Carbon Steel and Alloy Steel Welded Fittings of Seamless or Welded Construction for Low-Temperature Service (A 420 - 61 T):

Section 10(a).—At the end of the paragraph, add the sentence: "Impact tests are not required for grades 304, 304L, and 347 for temperatures above −425 F."

Tentative Specification for Centrifugally Cast Ferritic Alloy Steel Pipe for High-Temperature Service (A 426 - 62 T):

New Section 3.—Add a new Section 3

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[6] This revised specification appears in the *1964 Book of ASTM Standards*, Part 3.

entitled "Basis of Purchase" to read as follows, renumbering the present sections accordingly:

3. Orders for material under this specification shall include the following, as required, to describe the desired material adequately:

(*1*) Quantity (feet or number of lengths),
(*2*) Name of material (centrifugally cast pipe),
(*3*) Grade (Table I),
(*4*) Size (inside diameter and minimum wall thickness),
(*5*) Length (specific or random, Section 18),
(*6*) End finish (Section 19),
(*7*) Optional requirements (check analysis, Section 7; special hydrostatic test, Section 11, Note 3; repair by welding, Section 20(*d*); supplementary requirements S1 to S7),
(*8*) Test report required (Section 22(*b*)),
(*9*) ASTM designation, and
(*10*) Special requirements or exceptions to specification.

Section 11(a).—Change definitions of "*t*" and "*D*" as indicated above for Section 12(*a*) of A 155.

Tentative Specification for General Requirements for Carbon, Ferritic Alloy, and Austenitic Alloy Steel Tubes (A 450 – 62 T):

Section 10.—In the heading for this section delete the words "for Welded Tubing." This change permits seamless tubing to be examined, in addition to welded tubing, by the nondestructive electric test.

Add a new sentence to read: "Presently recognized methods capable of detecting the defect sizes detailed in Table IV include electromagnetic (eddy current), ultrasonic, and magnetic particle tests, the method chosen being dependent on the type of metal and size of tube being tested.

Tentative Specification for Centrifugally Cast Austenitic Steel Pipe for High-Temperature Service (A 451 – 62 T):

New Section 3.—Add a new Section 3 entitled "Basis of Purchase" to read as follows, remembering the present sections accordingly:

3. Orders for material under this specification shall include the following, as required, to describe the desired material adequately:

(*1*) Quantity (feet or number of lengths),
(*2*) Name of material (centrifugally cast pipe),
(*3*) Grade (Table II),
(*4*) Size (outside or inside diameter and minimum wall thickness),
(*5*) Length (specific or random, Section 16),
(*6*) End finish (Section 17),
(*7*) Optional requirements (check analysis, Section 7; special hydrostatic test, Section 10, Note 1; repair by welding, Section 18(*c*); supplementary requirements S1 to S7),
(*8*) Test report required (Section 20(*a*)),
(*9*) ASTM designation, and
(*10*) Special requirements or exceptions to specification.

Section 10(a).—Change definitions of "*t*" and "*D*" as indicated above for Section 10 of Specification A 106.

Tentative Specification for Centrifugally Cast Austenitic Cold-Wrought Pipe for High-Temperature Service (A 452 – 62 T):

New Section 3.—Add a new Section 3 entitled "Basis of Purchase" to read as follows, renumbering the present sections accordingly:

3. Orders for material under this specification shall include the following, as required, to describe the desired material adequately:

(*1*) Quantity (feet or number of lengths),
(*2*) Name of material (centrifugally cast cold-wrought pipe),
(3) Grade (Table I),
(*4*) Size (inside diameter and minimum wall thickness),
(*5*) Length (specific or random, Section 17),
(*6*) End finish (Section 18),
(*7*) Optional requirements (check analysis, Section 8; special hydrostatic test, Section 11, Note 2; repair by welding, Section 19(*c*); supplementary requirements S1 to S7),
(*8*) Test report required (Section 21(*c*)),
(*9*) ASTM Designation, and
(*10*) Special requirements or exceptions to specification.

Section 12(a).—Change definitions of "*t*" and "*D*" as indicated above for Section 10 of Specification A 106.

Tentative Method for Ultrasonic Testing and Inspection of Turbine and Generator Steel Rotor Forgings (A 418 – 57 T):

This method has been extensively revised and is appended hereto in its proposed revised form.[2]

A new Section 3 on "Basis of Purchase" similar to those outlined above in steel tubular specifications is to be added to the following specifications:

Tentative Specifications for:

Seamless Carbon Steel Boiler Tubes (**A 83 – 62 T**),
Seamless Low-Carbon and Carbon-Molybdenum Steel Still Tubes for Refinery Service (**A 161 – 62 T**),
Electric-Resistance-Welded Carbon Steel Boiler Tubes (**A 178 – 62 T**),
Seamless Cold-Drawn Low-Carbon Steel Heat-Exchanger and Condenser Tubes (**A 179 – 62 T**)
Seamless Carbon Steel Boiler Tubes for High-Pressure Service (**A 192 – 62 T**),
Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes (**A 199 – 62 T**),
Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service (**A 200 – 62 T**),
Seamless Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (**A 209 – 62 T**),
Seamless Medium-Carbon Steel Boiler and Superheater Tubes (**A 210 – 62 T**),
Seamless Ferritic and Austenitic Alloy-Steel Boiler, Superheater, and Heat Exchanger Tubes (**A 213 – 62 T**),
Electric-Resistance-Welded Carbon Steel Heat-Exchanger and Condenser Tubes (**A 214 – 62 T**),
Electric-Resistance-Welded Carbon Steel Boiler and Superheater Tubes for High-Pressure Service (**A 226 – 62 T**),
Welded Austenitic Steel Boiler, Superheater, Heat Exchanger, and Condenser Tubes (**A 249 – 62 T**),
Electric-Resistance-Welded Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (**A 250 – 62 T**),
Seamless and Welded Ferritic Stainless Steel Tubing for General Service (**A 268 – 62 T**),
Seamless and Welded Austenitic Stainless Steel Tubing for General Service (**A 269 – 62 T**),
Seamless and Welded Austenitic Stainless Steel Sanitary Tubing (**A 270 – 62 T**),
Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (**A 271 – 62 T**),
Ferritic Alloy Steel Forged and Bored Pipe for High-Temperature Service (**A 369 – 62 T**),
Seamless and Electric Welded Low Alloy Steel Tubes (**A 423 – 62 T**), and
Austenitic Steel Forged and Bored Pipe for High-Temperature Service (**A 430 – 62 T**).

Revision of Standards and Reversion to Tentative

Standard Specification for Hard-Drawn Steel Spring Wire (A 227 – 47),

Standard Specification for Steel Music Spring Wire (A 228 – 51),

Standard Specification for Oil-Tempered Steel Spring Wire (A 229 – 56),

Standard Specification for Carbon-Steel Valve Spring Quality Wire (A 230 – 47),

Standard Specification for Chromium-Vanadium Steel Spring Wire (A 231 – 41),

Standard Specification for Chromium-Vanadium Steel Valve Spring Quality Wire (A 232 – 47), and

Standard Specification for Chromium-Silicon Steel Spring Wire (A 401 - 58):

These specifications have been extensively revised and are appended hereto in their proposed revised form.[6]

Revision of Standards, Immediate Adoption

(requires nine-tenths affirmative vote)

Standard Specification for Low-Carbon Steel Joint Bars (A 3 – 54):

Section 1.—Change to read: "These specifications cover low-carbon steel joint bars primarily for industrial and mine use."

Section 6.—Change the requirement

for tensile strength from "55,000 to 65,000" to read "55,000 min."

Standard Specification for Medium-Carbon Steel Joint Bars (A 4 – 54):

Section 1.—Change to read "These specifications cover medium-carbon steel joint bars primarily for industrial and mine use."

Standard Specification for Electric-Fusion (Arc)-Welded Steel Plate Pipe (A 134 – 62):

Section 7(a).—Change the definition of "t" and "D" to read:

t = specified wall thickness, in inches, and
D = specified outside diameter, in inches.

Standard Specification for Electric-Fusion (Arc)-Welded Steel Pipe (A 139 – 62):

Section 10(a).—Change as indicated above for Section 7(*a*) of Specification A 134.

Standard Specification for Spiral-Welded Steel or Iron Pipe (A 211 – 54):

Section 4.—Change as indicated above for Section 7(*a*) of Specification A 134.

Standard Specification for Metal-Arc Welded Steel Pipe for High-Pressure Transmission Service (A 381 – 61):

Section 8(a).—Change as indicated above for Section 7(*a*) of Specification A 134.

REPORT OF COMMITTEE A-3

ON

CAST IRON*

Committee A-3 on Cast Iron held two meetings during the year: in New York, N. Y., on June 29, 1962, and in Detroit, Mich., on Jan. 25, 1963. A number of subcommittees and task groups met in conjunction with the main committee, and also between meetings of the main committee.

The committee consists of 132 members, of whom 114 are voting members; 41 are classified as producers, 37 as consumers, and 36 as general interest members. There are 2 consulting members.

The committee lost through death a long-time valued member, H. A. Bright.

A new task group on abrasion-resistant irons was formed under the chairmanship of G. H. Davis. The task groups on thermal fatigue, on sampling and analysis, and on compilation of standards on cast iron, were disbanded. D. E. Krause resigned as chairman of the task group on International Standards and H. W. Stuart was appointed to replace him.

During the year the Compilation of ASTM Standards on Iron Castings was published.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1962 Annual Meeting, Committee A-3 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specification for:

Nodular Iron Castings for Paper Mill Dryer Rolls (A 476 – 62 T).

Revision of Tentative Specification for:

Gray Iron Castings for Pressure-Containing Parts for Temperatures Up to 650 F (A 278 – 61 T).

These recommendations were accepted by the Administrative Committee on Sept. 11, 1962, and the new and revised tentatives appear in the 1962 Supplement to Book of ASTM Standards, Part 1.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee A-3 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] The existing tentatives referred to in this report appear in their present form in the 1961 Book of ASTM Standards, Part 1.

Revision of Tentative

Tentative Specifications for Ferritic Nodular Iron Castings for Valves,

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* **Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.**

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Flanges, Pipe Fittings, and Other Piping Components (**A 445 – 60 T**): Revise as appended hereto.[2]

Adoption of Tentative as Standard Without Revision

Tentative Specification for Austenitic Gray Iron Castings (**A 436 – 61 T**).

Tentative Continued Without Revision

Tentative Specifications for Gray Iron Castings for Valves, Flanges, and Pipe Fittings (**A 126 – 61 T**):

This specification is recommended for continuance as tentative pending a study between Subcommittees 17 and 6 on the possible development of an agreement under Specifications A 126 concerning a test to be recognized under Specifications A 48 when transverse testing is necessary.

Standards Continued Without Revision

All of the standards under the jurisdiction of Committee A-3 that have stood six years or more without revision are still considered in accord with present practice and require no revision, except as noted in the subcommittee reports.

ACTIVITIES OF SUBCOMMITTEES

Task Group on International Standards (H. W. Stuart, chairman).—The resignation of D. E. Krause was accepted with sincere appreciation for his fine work, and H. W. Stuart was appointed to be interim chairman until a key man within Committee A-3 is developed who will permanently take the responsibility for this work. The task group will continue to maintain liaison with ISO Technical Committee 25 in cooperation with the ASA staff.

Task Group on Abrasion-Resistant Irons (G. H. Davis, chairman).—The assignment of this task group was to show why Committee A-3 should or should not prepare a specification for abrasion-resistant irons. After considerable discussion the task group expressed the unanimous opinion that there is a need for specifications covering abrasion-resistant cast irons and that Committee A-3 should undertake the task of preparing such specifications.

Task Group on Sampling and Analysis (George Krumlauf, chairman) was disbanded with thanks because its assignment had been successfully completed.

Task Group on Compilation of Standards on Cast Iron (H. W. Lownie, Jr., chairman) was disbanded with thanks because its assignment had been successfully completed.

Task Group on Thermal Fatigue (W. Spear, chairman) was disbanded because of lack of interest.

Subcommittee 6 on General Castings (H. W. Lownie, Jr., chairman) is working on a proposed revision of Specifications A 48 consisting of the addition of a class 55 iron. The major current activity is entrusted to a subcommittee task group under the chairmanship of Fred Sefing, which is to recommend to Subcommittee 6 a new product specification for thin-section castings of uniform and reliable physical characteristics.

Subcommittee 7 on Microstructure of Cast Iron (C. K. Donoho, chairman) is working on a recommended practice for evaluating the microstructure of nodular iron. Acceptance of this recommended practice has been delayed because of a divergence of opinion concerning methods to measure and specify sizes of graphite particles, but there appears to be reasonable concurrence on designations for forms of graphite in nodular iron. Consequently, the subcommittee plans to submit to Committee A-3 before the 1963 Annual Meeting a recommended practice for evaluating the form of graphite in nodular iron. A recommendation for evaluating graphite size will be delayed pending further analysis of the problem.

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[2] The revised tentative appears in the *1964 Book of ASTM Standards*, Part 2.

Subcommittee 11 on Methods of Test (W. E. Gruver, chairman) has been assigned to the task of preparing a recommended practice for impact testing of nodular iron. The need for such a method has become apparent from the large demand for impact data among consumers of nodular iron and competitive materials. Subcommittee 11 is also considering the need for a method to determine the physical properties of sections less than $\frac{3}{16}$ in. thick.

Subcommittee 16 on Heat Treatment of Cast Iron (T. E. Eagan, chairman) is preparing recommended practices for: (1) stress-relief annealing of all types of cast iron (except malleable iron), (2) annealing of all types of cast iron (except malleable iron), and (3) quenching and tempering practices for all types of cast iron (except malleable iron).

Subcommittee 17 on Cast Iron Valves, Flanges, and Fittings (J. J. Kanter, chairman) has completed and received committee approval for revision of Specification A 445. The revision, as appended to this report, permits qualification of valves and fittings of 3-in. pipe size and smaller by microstructural examination when agreed by supplier and purchaser.

Subcommittee 18 on Automotive Castings (R. A. Clark, chairman) is attempting to reconcile the differences between Specification A 159 and the corresponding SAE standard "Gray Iron for Automotive Castings." To accomplish this, the Subcommittee intends to recommend revisions to both SAE and ASTM.

Subcommittee 20 on Soil Pipe and Fittings (J. W. Perry, Jr., chairman) has been revived after nearly 20 years of inactivity. The main activity is the preparation of a revision of the Standard Specification for Cast Iron Soil Pipe and Fittings (A 74) which will include complete data on dimensions, tolerances, weights, chemical requirements, and physical tests. This revision is needed because the present specification is out of date.

Subcommittee 21 on Pressure Pipe (J. T. Vann, chairman) is responsible for the reference specification A 377 that lists the American Standards covering cast iron pressure pipe. The American Standards are now in the process of revision, and when these revisions have been completed, Subcommittee 21 will review the standards and recommend action on Specification A 377.

Subcommittee 22 on Elevated-Temperature Properties of Cast Iron (J. R. Kattus, chairman) has recently completed a revision of the Tentative Specification for Gray Iron Castings for Pressure-Containing Parts for Temperatures Up to 650 F (A 278) to include class 70 and 80 irons. This revision was accepted by the Administrative Committee on Standards. At present a task group under J. S. Vanick is considering the possibility of preparing a specification for acicular irons. The ASME Pressure Vessel Committee has requested such a specification to eventually supersede Code Case 1303, "Use of Acicular Iron for Unfired Pressure Vessels."

Subcommittee 24 on Nodular Iron (H. W. Ruf, chairman) has completed the new Tentative Specification for Nodular Iron Castings for Paper Mill Dryer Rolls (A 476) which was accepted by the Administrative Committee on Standards. A survey of the mechanical properties of nodular iron is being conducted by a task force under Don Crews. A summary of the data and recommendations will be submitted at the next meeting.

Subcommittee 25 on Chill Testing of Cast Iron (George Krumlauf, chairman) plans to review the method for chill testing (A 367) and to consider the advisability of preparing a standard for the carbon-equivalent test.

This report has been submitted to a letter ballot of the committee, which consists of 114 voting members; 75 members returned their ballots, of whom 70 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

H. W. LOWNIE, JR.,
Chairman.

J. R. KATTUS,
Secretary.

REPORT OF COMMITTEE A-5
ON
CORROSION OF IRON AND STEEL*

Committee A-5 on Corrosion of Iron and Steel held two meetings during the year: on June 27, 1962, in New York, N.Y., and on Jan. 22, 1963, in Detroit, Mich. Most of the subcommittees held meetings prior to the main committee meetings.

The committee consists of 108 members, of whom 50 are classified as producers, 33 as consumers, and 25 as general interest members.

One of the subcommittee chairmen resigned due to a change in employment, and Miss Jane H. Rigo was appointed chairman of Subcommittee XV to fill the vacancy, and John R. Wellington was appointed chairman of Subcommittee VII to replace Miss Rigo.

The following committee members were elected to serve on the Advisory Committee as members-at-large: W. C. Clements, G. A. Ellinger, H. F. Hormann, F. A. Webber, and C. P. Larrabee.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1962 Annual Meeting, Committee A-5 presented to the Society through the Administrative Committee on Standards the following recommendations, which were accepted by the Standards Committee on the dates indicated:

New Tentative Specifications for:

Aluminum Coated Steel Wire Strand (A 474 – 62 T), and

Zinc-Coated Steel Wire Strand (A 475 – 62 T). This specification replaces Specifications A 122 and A 218 which were therefore withdrawn.

Revision of Tentative Specifications for:

Long Terne Iron or Steel Sheets, Coils and Cut Lengths (A 308 – 59 T),

Zinc-Coated (Galvanized) Iron or Steel Sheets, Coils and Cut Lengths (A 93 – 59 T),

Zinc-Coated (Galvanized) Iron or Steel Sheets for Culverts and Underdrains (A 444 – 60 T),

Zinc-Coated (Galvanized) Steel Sheets of Structural Quality, Coils and Cut Lengths (A 446 – 60 T)

1.25 Oz Class Coating (Pot Yield) Zinc-Coated (Galvanized) Iron or Steel Roofing Sheets (A 361 – 59 T)

The new Tentative Specifications A 474 and A 475 were accepted by the Standards Committee on July 27, 1962, and appear in the 1962 Supplement to Book of ASTM Standards, Part 1. The revision of Tentative Specification A 361 was accepted on April 29, 1963, and the revisions of the other specifications were accepted on March 21, 1963.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee A-5 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] All of the existing tentatives and standards referred to appear in their present form in the 1961 Book of ASTM Standards, Part 1, or the 1962 Supplement.

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

New Tentative

Tentative Specification for Aluminum-Coated Steel Chain-Link Fence Fabric, as appended hereto.[2]

Revision of Tentative

Tentative Specification for Zinc-Coated Steel Chain-Link Fence Fabric (**A 392 – 59 T**):
Revise as appended hereto.[2]

Revision of Standard, Immediate Adoption

(requires nine-tenths affirmative vote)

Standard Specification for Zinc (Hot-Galvanized) Coatings on Products Fabricated from Rolled, Pressed, and Forged Steel Shapes, Plates, Bars, and Strip (**A 123 – 59**):

Section 1(b).—Delete the last sentence as not being appropriate for inclusion in a specification for galvanizing.

Adoption of Tentative as Standard Without Revision

Tentative Specification for Zinc-Coated Flat Steel Armoring Tape (**A 459 – 61 T**).

Tentatives Continued Without Revision

Tentative Method of Test for:

Weight of Coating on Aluminum-Coated Iron or Steel Articles (**A 428 – 58 T**). A study is under way on a chemical means for weight determination.

Tentative Specification for:

Copper-Covered Steel Wire Strand (**A 460 – 61 T**). A review is in progress on the classes of coating thickness.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee VII on Methods of Testing (J. R. Wellington, chairman) is continuing its investigation on uniformity tests for aluminized steel wire and weight determinations of aluminum coatings. The committee has recognized the growing interest in the use of electronic gages for determining coating thickness and is initiating cooperation with other ASTM technical committees for exchange of information.

Subcommittee XI on Sheet Specifications (E. P. Beachum, chairman) is revising the Specification for Zinc-Coated Iron or Steel Sheets, Coils, and Cut Lengths (A 93) to include general purpose provisions. Several new specifications are also under way to broaden the coverage of coated sheet specifications. A task force has recently been appointed to investigate the feasibility of reporting metallic coatings on steel sheets in terms of thickness as well as weight.

Subcommittee XII on Wire Specifications (J. F. Occasione, chairman) prepared the two new tentative specifications which were approved by the Administrative Committee on Standards: on Aluminum-Coated Steel Wire Strand (A 474) and on Zinc-Coated Steel Wire Strand (A 475). A proposed tentative specification for aluminum-coated steel chain-link fence fabric and a proposed revision of the Specification for Zinc-Coated Steel Chain-Link Fence Fabric (A 392) have been submitted to Committee A-5.

Subcommittee XIII on Hardware Specifications (R. A. Munster, chairman) will continue to study the premature discoloration and corrosion of the zinc coating on transmission tower steel with the possibility of preparing a specification covering the various steels that are now normally being galvanized, and the precautions to be taken during galvanizing to get better coatings on these steels. Two task groups have been appointed: one to prepare a list of technical terms and their meanings, and the other to study and revise the hammer test requirements of the Specification A 123.

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[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[2] The new and revised Specifications appear in the *1964 Book of ASTM Standards*, Part 3.

Subcommittee XIV on Sheet Inspections (O. B. Ellis, chairman) inspected the corrugated metallic-coated sheets which were exposed at five locations in 1960, and the corrugated galvanized and black sheets which were exposed at State College, Pa., in 1926. Reports of the seven inspections made in 1961 and 1962 are appended to this report.[3]

Subcommittee XV on Wire Tests (Miss J. H. Rigo, chairman) exposed fabricated and unfabricated aluminized steel wire specimens in 1961 at seven test sites in the United States. The exposure program in England is temporarily delayed by the inability of the cooperating company to locate and purchase a suitable test site. The committee is presently engaged in preparing a summary report covering the scope and experimental details of the aluminized steel wire test program. Inspection of the galvanized wire test program continues and a status report is planned for late 1963.

Subcommittee XVI on Hardware Tests (L. E. Fiorito, chairman) inspected the specimens which were exposed at the three test sites in 1958. Reports of the field tests on atmospheric corrosion are appended to this report.[4] The subcommittee also inspected the hardware of the 1929 program remaining at State College, Pa., on April 30, 1962. In accordance with custom, this report will be included in next year's committee report since the changes are not very significant. An investigation is under way on the 1929 program to evaluate the necessity for continuing this exposure.

This report has been submitted to letter ballot of the committee, which consists of 108 members; 87 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

C. W. STRAITOR,
Chairman.

S. K. COBURN,
Secretary.

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[3] See p. 132.

[4] See p. 136.

APPENDIX I

REPORT OF SUBCOMMITTEE XIV ON FIELD TESTS OF ATMOSPHERIC CORROSION OF METALLIC-COATED STEEL PANELS

1960 PROGRAM

The weights of coating of sheets exposed in 1960 were given in Appendix I to the 1961 Report of Committee A-5: Report of Subcommittee XIV on Field Tests of Atmospheric Corrosion of Metallic-Coated Steel Panels, Table I. Inadvertently coating weights for two materials, KL2 and QL1, were omitted. The corrected tabulation is shown in the accompanying Table I.

TABLE I.—IDENTIFICATION AND WEIGHTS OF COATING ON SHEETS IN TEST OF METALLIC-COATED STEELS.
1960 PROGRAM

Code	Coating Type	Ounces Per Square Foot				
		State College	Newark	Kure Beach	Point Reyes	Brazos River
AP1	Pot[a]	1.05	1.02	1.03	1.02	1.01
EP1	Pot	1.38	1.41	1.39	1.40	1.40
RP1	Pot	1.06	1.06	1.06	1.05	1.03
JP2	Pot[a]	2.43	2.42	2.39	2.37	2.40
PP2	Pot	2.25	2.24	2.21	2.27	2.28
CL1	Line[b]	1.17	1.19	1.16	1.14	1.18
GL1	Line	1.21	1.21	1.21	1.21	1.20
IL1	Line	1.18	1.20	1.20	1.18	1.18
LL1	Line	1.05	1.06	1.01	0.99	1.00
OL1	Line	1.06	1.06	1.06	1.07	1.05
QL1	Line	1.16	1.15	1.15	1.15	1.14
KL2	Line[b]	2.51	2.41	2.32	2.43	2.47
BL2	Line	2.06	1.98	1.86	2.03	2.17
HL2	Line	2.12	2.12	2.12	2.13	2.14
ML2	Line	2.92	3.00	3.09	3.06	3.02
NL2	Line	1.82	1.83	1.88	1.92	1.94
SA1	Al-Si[c]	0.91	0.92	0.93	0.92	0.90
TA1	Al[d]	1.38	1.39	1.38	1.39	1.42
UT4	Terne[e]	1.28	1.31	1.31	1.29	1.34

[a] Individually dipped in conventional galvanizing pots.
[b] Continuous galvanizing line.
[c] Aluminum-silicon coated sheets.
[d] Pure aluminum coated sheets.
[e] Terne-alloy coated sheets.

The atmospheric corrosion tests of corrugated metallic-coated steel panels exposed in 1960 were inspected as shown below:

State College, Pa.	April 24, 1961 and April 30, 1962
Newark, N. J.	May 1, 1962 and October 18, 1962
Kure Beach (800-ft site), N. C.	Aug. 23, 1962
Brazos River (Freeport), Tex.	Sept. 11, 1962
Point Reyes, Calif.	Sept. 13, 1962

TABLE II.—ATMOSPHERIC CORROSION OF CORRUGATED METALLIC-COATED STEEL PANELS.

Exposed September 1960

Coating Type	Code	Average Wt. Coating, oz per ft^2	State College, 1.6 yr.	Newark, 2.1 yr.	Kure Beach, 1.9 yr	Point Reyes, 2 yr.	Brazos River, 2 yr.
Galvanized—Pot	RP1	1.05	G	2% Y	TrA	TrA	0.1A[a]
Galvanized—Pot	EP1	1.40	G	8% A	G	G	G
Galvanized—Pot	AP1	1.03	G	TrY	G	G	G[a]
Galvanized—Pot	JP2	2.40	G	G	G	G	G
Galvanized—Pot	PP2	2.25	G	25% A	G	G	G
Galvanized—Line	CL1	1.17	G	G	G	G	G
Galvanized—Line	GL1	1.21	G	G	G	G	G
Galvanized—Line	IL1	1.19	G	G	G	G	G
Galvanized—Line	LL1	1.02	G	G	G	G	G
Galvanized—Line	OL1	1.06	G	TrY	G	G	G
Galvanized—Line	QL1	1.15	G	G	G	G	G
Galvanized—Line	KL2	2.43	G	G	G	G	G
Galvanized—Line	HL2	2.13	G	G	G	G	G
Galvanized—Line	BL2	2.02	G	G	G	G	G
Galvanized—Line	ML2	3.02	G	G	G	G	G
Galvanized—Line	NL2	1.88	G	G	G	G	G
Aluminum Silicon	SA1	0.92	35 phr	TrY	OK	OK[a]	OK[a]
Aluminum	TA1	1.39	Tr. Edge Stain	OK	OK	OK	12 phr
Terne	UT4	1.31	Severe phr	Slight phr	Severe phr	15% phr	50% phr[a]

A—Zinc-Iron alloy visible.
G—Continuous zinc surface covered with corrosion products of zinc.
Tr—Trace.
Y—Yellow corrosion products of alloy.
R—Base metal rust.
phr—Pinhole rust spots, surrounded by brown stain.
[a] Sheet cracked at 1 or more bolt holes.

A summary of the results of inspections is given in Table II. At all test sites most of the zinc-coated sheets were covered with a relatively uniform accumulation of zinc corrosion products. One or more of the 1.25-oz coating class pot-galvanized sheets showed dark areas of iron-zinc alloy except at State College.

At Newark three sheets, RP1, AP1, and OL1, had developed small amounts of yellow rust.

The aluminum-coated sheets showed only superficial corrosion as evidenced by a few pinhole rust spots at Brazos River and State College, slight edge stain at State College and some yellow-

TABLE III.—LIFE OF CORRUGATED GALVANIZED AND BLACK SHEETS, 26 BY 30 IN.

Exposed at State College, Pa., on April 23, 1926.

Boldface numbers indicate changes since 1961 Report.

Sheet[b]	No. 16 Gage, 2.5 oz per sq ft[a]		No. 22 Gage, 2.5 oz per sq ft[a]			No. 22 Gage, 2.0 oz per sq ft[a]			No. 22 Gage, 1.5 oz per sq ft[a]			No. 22 Gage, 1.25 oz per sq ft[a]			No. 22 Gage, 0.75 oz per sq ft[a]			No. 28 Gage, 0.75 oz per sq ft[a]			No. 22 Gage Uncoated
	Years to		Years to			Years to			Years to			Years to			Years to			Years to			
	1st Rust	100 per cent Rust	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	Years to Perforation
R (T)	24.0	*	26.0	*	*	19.0	*	*	17.5	*	*	14.5	**36.0**	*	[c]			[c]			26.0
(B)	27.0	*	29.0	*	*	20.0	*	*	16.5	*	*	15.0	*	*	[c]			[c]			
V (T)	27.0	*	21.5	*	*	18.5	*	*	16.0	*	*	15.0	*	*	9.5	23.0	*	[c]			24.0(V)
(or U)(B)	24.0	*	29.0	*	*	25.0	*	*	15.5	*	*	16.5	*	*	10.5	24.0	*	[c]			25.0(U)
N (T)	[c]		20.5	*	*	22.5	*	*	17.5	*	*	12.0	**36.0**	*	6.5	20.5	*	11.0	23.0	*	28.0
(B)	[c]		28.0	*	*	22.5	*	*	15.5	*	*	13.5	**36.0**	*	9.0	23.0	*	11.5	30.0	[d]	
T (T)	27.0	*	34.4	*	*	21.0	*	*	16.0	*	*	15.0	*	*	9.0	26.0	*	9.0	24.0	*	*
(B)	31.0	*	*	*	*	23.0	*	*	14.5	*	*	13.5	34.4	*	10.5	22.5	*	12.0	25.0	*	
Y (T)	21.5	*	22.5	*	*	19.5	*	*	17.5	*	*	14.5	*	*	10.0	22.5	*	9.5	24.0	*	25.0
(B)	22.5	*	29.0	*	*	19.0	*	*	18.5	*	*	14.5	29.0	*	10.5	30.0	*	13.0	26.0	*	
S (T)	22.0	*	28.0	*	*	25.0	*	*	19.0	*	*	14.5	32.0	*	10.5	21.5	*	11.5	22.0	*	*
(B)	22.5	*	32.0	*	*	28.0	*	*	19.5	*	*	15.5	30.0	*	11.0	21.5	*	11.5	21.5	*	
C (T)	[c]		22.5	*	*	17.5	*	*	16.0	*	*	14.5	*	*	9.5	24.0	*	[c]			*
(B)	[c]		34.4	*	*	21.5	*	*	18.5	*	*	14.5	30.0	*	10.0	24.0	*	[c]			
K (T)	23.0	*	25.0	*	*	22.5	*	*	17.5	*	*	14.5	31.0	*	11.5	24.0	*	10.5	23.0	*	*
(B)	24.0	*	30.0	*	*	28.0	*	*	21.0	*	*	16.5	33.0	*	11.5	22.5	*	11.5	21.0	*	
W (T)	[c]		29.0	*	*	25.0	*	*	16.5	33.0	*	13.5	31.0	*	[c]			[c]			27.0
(B)	[c]		**36.0**	*	*	28.0	*	*	15.0	35.0	*	14.5	30.0	*	[c]			[c]			
Average	24.6	...	...	...	...	22.5	...	...	17.1	...	...	14.6			10.0	23.5		11.1	24.0		

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* Data not yet available.

[a] Coating weight is "oz per sq ft" of sheet (both sides).

[b] (T) = Corrugated sample from top of sheet on west side of rack with bottom surface upward.
(B) = Corrugated sample from bottom of sheet on east side of rack with top surface upward.

[c] There were no specimens exposed.

[d] Reported as perforation rather than 100 per cent rust in 1956 *Proceedings*.

ish corrosion on crests of corrugations at Newark.

The terne-coated sheet, UT4, developed pinhole rusting at all exposure sites. The degree of rusting ranged from slight in the industrial atmosphere at Newark to severe at all other test sites.

At Brazos River and Point Reyes, some sheets had cracked at bolt holes. These were light gage sheets, and it appeared probable that vibration during wind storms had caused the damage. If the cracking becomes worse, it is doubtful that the sheets can be satisfactorily secured to the test racks.

1926 PROGRAM

At the 1961 inspection of galvanized sheets in the 1926 test at State College, one sheet, WCK(B), in the 1.5-oz coating class was found to be 100 per cent rusted. In 1962 WAE(B) in the 2.5 oz coating class was a new coating failure. In the 1.25 oz coating class sheets RET(T), NEU(T) and NEU(B) were 100 per cent rusted. The complete tabulation is given in Table III.

Respectfully submitted on behalf of Subcommittee XIV.

O. B. ELLIS,
Chairman.

APPENDIX II

REPORT OF SUBCOMMITTEE XVI ON FIELD TESTS OF ATMOSPHERIC CORROSION OF HARDWARE

This report covers the 1962 inspections of the 1958 program at Newark, N. J., and at Kure Beach, N. C.

Background and details—including basis metal compositions, coating procedures, individual coating thicknesses, and microscopic examinations—are in the 1959 ASTM *Proceedings*, p. 133. In the 1960 ASTM *Proceedings*, p. 116, tabulations of the ranges and averages of thickness are condensed for all sets of specimens.

The samples for this test program were prepared to meet a specified coating thickness of 2.0 to 2.5 mils, which was established by the Subcommittee to keep the time of exposure to a reasonable length.

A table in the 1961 ASTM *Proceedings*, p. 156, shows the weight loss data for uncoated panels of six of the basis steels removed from the three sites, after approximately 2-yr exposure. These weight losses of the basis steels reflect an initially high corrosion rate which, particularly in the case of the low-alloy steels, becomes much reduced with increased exposure. The subcommittee has approved the removal of additional specimens at the end of 5 and 10 yr for further weight-loss data.

In the accompanying Table I is the inspection data for the Newark, N. J., site specimens. The numbering system for specimen identification is shown in Fig. 2 of the report in the 1959 ASTM *Proceedings*, p. 133. In this system, a specimen is identified by four numbers, for example, 0-5-11-14, where the first number represents the basis metal, the second the coating, the third the exposure location, and the last is one of five replicate numbers.

NEWARK SITE

Due to the weather conditions in 1962, some specimens appear to have improved in appearance, when the report is compared with that in 1961. Obviously, this cannot be true, so an absence of TrY at this time, while it was noted in 1961, should be considered as of little consequence.

Only specimen 1-8-11-16 shows any degree of rust. A few hot-dip zinc specimens (code 5) show spots or trace of rust but none exceed 1 per cent. Specimen 0-5-11-15 was removed because of damage. All of these hot-dip zinc specimens show signs of dark discoloration, varying from a trace to as much as 90 per cent on a surface (top or bottom). This was first noted in 1961 by the inspectors and is considered more pronounced in 1962. No conclusions have been drawn.

All hot-dip aluminum specimens (codes 6 and 7) exhibit a general darkening on the surfaces of all flat and bent panels. Only a TrR has been noted on four specimens, which, however, is evident at only the edges of the bends.

Of the electroplated zinc specimens (code 8) two show TrR and one (1-8-11-

TABLE I.—REPORT OF INSPECTION OF COATED STEEL HARDWARE, NEWARK, N. J. (CODE 11).

Exposed: 6-13-58; inspected: 5-1-62; exposure period: 3.93 years.

Abbreviations and symbols: 0 = no rust; 5, 10, 25, 100 = approximate percentages of rust; R = rusting of base metal; Y = yellow or orange rust; Tr = trace; N = no specimen exposed; $\bar{X}$ = average measured coating thickness.

Specimen	Flat Panels			Bent Panels			Rounds	
Steel Coating Location Replicate	$\bar{X}$ Mils	Topside	Underside	$\bar{X}$ Mils	Topside	Underside	$\bar{X}$ Mils	Sides
Hot-dip zinc (code 5)[b]								
Carbon steel (code 1)								
1-5-11-14	2.44	TrY	0	2.28	...[c]	0	...[a]	...
1-5-11-17	1.86	TrR, 5Y[d]	0	1.91	TrY	0	...	...
1-5-11-18	2.70	TrY	0	...	...	...	...	...
Low-alloy steels I, II, III, and IV (codes 2, 3, 4, and 0 (no code))								
2-5-11-14	3.66	TrY	0	2.34	5Y	TrY	...	...
2-5-11-15	2.81	TrY	0	2.46	TrY	TrY	...	...
2-5-11-16	3.14	TrY	0	2.29	TrY	TrY	...	...
2-5-11-17	3.08	TrY	0	2.60	TrY	0	...	...
2-5-11-18	3.74	TrY	0	2.28	5Y	0	...	...
3-5-11-14	2.63	TrY	0	2.42	5Y	0	...	N
3-5-11-15	2.54	TrY	0	2.41	5Y	TrY	...	N
3-5-11-16	2.86	TrY	0	2.72	TrY	0	...	N
3-5-11-17	3.18	TrY	0	2.50	TrY	0	...	N
3-5-11-18	2.72	TrY	0	2.49	5Y	0	...	N
4-5-11-14	4.11	TrY	0	...	...	...	...	N
4-5-11-15	3.31	TrY	0	...	...	...	...	...
4-5-11-16	4.19	TrY	TrR[d]	...	...	...	...	...
0-5-11-14	3.29	TrY	0	2.01	TrY	0	...	...
0-5-11-16	2.92	TrY	0	2.18	TrY	0	...	...
0-5-11-17	3.23	TrY	0	2.35	TrY	0	...	...
0-5-11-18	3.01	TrY	0	2.28	TrY	0	...	...
Nodular iron (cast: code 19)								
19-5-11-17	4.81	TrY	0	...	N	N	...	N
19-5-11-18	4.36	TrY	0	...	N	N	...	N
Malleable iron (code 21)								
21-5-11-14	7.23	TrY	0	...	N	N	...	N
21-5-11-15	7.06	TrY	0	...	N	N	...	N
21-5-11-16	7.36	TrY	0	...	N	N	...	N
21-5-11-17	6.89	TrY	0	...	N	N	...	N
21-5-11-18	7.43	5Y	0	...	N	N	...	N
Hot-dip aluminum (code 6)[e]								
Carbon steel (code 1)								
1-6-11-18	...	...	...	2.58	0	TrR[f]	...	...
Low-alloy steels I, II, III, and IV (codes 2, 3, 4, and 0 (no code))								
2-6-11-17	2.06	TrY	0	...	...	...	...	...
3-6-11-14	2.40	TrY[g]	0	1.76	0	TrR[h]	...	N
4-6-11-14	...	...	...	2.39	TrR[i]	0	...	...
0-6-11-14	1.77	0	TrY	...	...	...	...	...
0-6-11-17	2.39	TrY	0	...	...	...	...	...
Hot-dip aluminum (code 7)[e]								
Carbon steel (code 1)								
1-7-11-15	...	...	...	...	...	...	3.11	TrY
1-7-11-16	...	...	...	...	...	...	3.26	TrY

TABLE I.—REPORT OF INSPECTION OF COATED STEEL HARDWARE, NEWARK, N. J. (CODE 11).—*Continued*

Specimen	Flat Panels			Bent Panels			Rounds	
Steel Coating Location Replicate	$\bar{X}$ Mils	Topside	Underside	$\bar{X}$ Mils	Topside	Underside	$\bar{X}$ Mils	Sides
Low-alloy steel I, III, and IV (codes 2, 4, and 0 (no code))								
2-7-11-18	...	...	...	...	...	...	3.24	TrY[j]
4-7-11-15	...	...	...	...	...	...	4.13	TrY[j]
4-7-11-18	...	...	...	2.32	0	TrR, TrY[k]	3.00	TrY
0-7-11-14	...	...	...	...	...	...	2.93	TrY
0-7-11-16	...	...	...	...	...	...	2.15	TrY
0-7-11-17	...	...	...	...	...	...	2.87	TrY
Electroplated zinc (code 8)								
Carbon steel (code 1)								
1-8-11-16	...	...	...	...	...	...	1.64	20R[l]
Low-alloy steel I (code 2)								
2-8-11-14	...	...	...	2.52	0	TrR[m]	...	...
2-8-11-15	...	...	...	...	...	...	2.29	TrR

[a] Specimens not showing a trace of yellow or rust are not included, hence the blank spaces in columns.

[b] Hot-dip zinc (code 5) samples were centrifuged immediately after coating to remove excess zinc. Details are contained in the 1959 ASTM *Proceedings*, p. 137.

[c] Two rust spots near edge at bend.

[d] One rust spot.

[e] Hot-dip aluminum samples (code 6) were prepared by immersing in the aluminizing bath for 1 min; code 7 samples were immersed for 2 min and rinsed in the fluxing bath immediately after coating, to remove excess aluminum. More complete details are found in the 1959 ASTM *Proceedings*, p. 137.

[f] On edge near bend.

[g] Around hole and upper right edge.

[h] Lower right edge and bend; may be due to mechanical damage.

[i] On edge and bend.

[j] At notch.

[k] At edge at corners and bends.

[l] 5R or more, first noted in 1962 during inspection after 3.93 yr of exposure.

[m] Top right corner.

16) shows 20 per cent R, as compared with 1 per cent R in 1961. All specimens exhibit a general darkening of the surfaces.

Only the sprayed zinc (code 9) and sprayed aluminum (code 10) specimens show no yellow or rust, except around identification holes, as noted previously. For the sprayed aluminum specimens, the flat panels exhibit generally dark spots speckled on the surfaces, and the bent panels occasionally show this speckling.

KURE BEACH, N. C.

800-ft Site:

Only the hot-dip aluminum specimens (code 7) show 5 per cent R or more rusting of the base metal. Of these 14 specimens, 4 are flat, 9 are bent, and 1 is round, a total of 5 more than were noted in 1961 or earlier. A few hot-dip aluminum specimens (code 6) show a trace of rust or low percentage of yellow. The sprayed aluminum specimens (code

TABLE II.—REPORT OF INSPECTION OF COATED STEEL HARDWARE, KURE BEACH, N.C. (800-FT SITE, CODE 12).

Exposed: 6-23-58; inspected: 6-5-62; exposure period: 3.94 years.

Abbreviations and symbols: 0 = no rust; 5, 10, 25, 100 = approximate percentages of rust; R = rusting of base metal; Y = yellow or orange rust; Tr = trace; N = no specimen exposed; $\overline{X}$ = average measured coating thickness.

Specimen	Flat Panels			Bent Panels			Rounds	
Steel Coating Location Replicate	$\overline{X}$ Mils	Topside	Underside	$\overline{X}$ Mils	Topside	Underside	$\overline{X}$ Mils	Sides
Hot-dip zinc (code 5)[a]								
Carbon Steel (Code 1)								
1-5-12-16	2.18	0	TrY	...[j]	...	...	...	...
1-5-12-17	...	[b]	...	...	...	...	...	...
1-5-12-18	2.01	TrY	0	...	...	...	...	...
Low-alloy steel III (code 4)								
4-5-12-16	2.74	TrY[c]	0	...	...	...	...	...
Nodular iron (cast: code 19)								
19-5-12-14	4.38	TrY	0	...	N	N	...	N
Nodular iron (machined: code 20)								
20-5-12-17	6.03	TrY	0	...	N	N	...	N
20-5-12-18	6.04	0	5Y	...	N	N	...	N
Hot-dip aluminum (code 6)[d]								
Carbon steel (code 1)								
1-6-12-14	...	...	...	2.48	5Y	TrR, 10Y	...	...
1-6-12-15	2.63	0	TrY[e]	2.55	TrY[e]	TrY	...	...
1-6-12-17	...	...	...	2.06	0	TrY	...	...
1-6-12-18	...	...	...	2.28	0	TrY	...	...
Low-alloy steel I, II, III, and IV (codes 2, 3, 4, and 0 (no code))								
2-6-12-14	2.16	TrY[c]	0	...	...	...	3.29	TrY[f]
2-6-12-15	2.06	0	TrY[c]	1.86	TrR	TrY	...	...
2-6-12-16	2.16	TrY	TrY[c]	1.94	0	TrY[c]	...	...
2-6-12-17	...	...	...	1.80	0	TrY[c]	...	...
2-6-12-18	...	...	...	1.99	0	TrY[c]	...	...
3-6-12-14	2.46	TrY[c]	TrY[c]	1.51	0	TrY[c]	...	N
3-6-12-15	2.17	0	TrY	1.42	0	TrY[c]	...	N
3-6-12-16	2.73	TrY[c]	0	1.96	0	TrY	...	N
3-6-12-17	2.43	TrR[c], TrY	TrR[c], TrY	...	...	...	...	N
3-6-12-18	2.43	TrY	TrY	1.45	0	TrY	...	N
4-6-12-14	2.38	TrY[e]	TrY[c]	2.22	0	TrY[c]	...	...
4-6-12-15	2.69	0	TrY	2.20	0	TrY	...	...
4-6-12-16	2.76	0	TrY	2.61	0	TrY[c]	...	...
4-6-12-17	...	...	...	1.89	TrY	5Y	...	...
4-6-12-18	2.79	0	TrY	2.21	TrY	TrR, 5Y	2.44	TrY[f]
0–6–12–15	2.32	TrY	TrY[e]	...	...	...	...	...
Hot-dip aluminum (code 7)[d]								
Carbon steel (code 1)								
1-7-12-14	...	...	...	2.49	0	5Y	...	...
1-7-12-15	3.79	TrR	0	2.34	0	5Y	...	...
1-7-12-16	...	...	...	2.86	0	TrY[c]	...	...
1-7-12-17	4.07	0	TrR[c]	2.60	0	TrR[c]	3.21	TrY[f]
1-7-12-18	3.80	0	TrR[c]	2.45	0	5Y	...	...
Low-alloy steel I, II, III, and IV (codes 2, 3, 4, and 0 (no code))								
2-7-12-14	3.33	30R, 20Y[g]	55R, 5Y[g]	...	...	...	...	...
2-7-12-15	...	...	...	2.28	0	TrY	...	...
2-7-12-16	3.79	40R, 20Y[g]	55R, 5Y[g]	2.96	TrY	10R, 5Y[i]	...	...

TABLE II.— REPORT OF INSPECTION OF COATED STEEL HARDWARE, KURE BEACH, N. C. (800-FT SITE, CODE 12).—*Continued*

Specimen	Flat Panels			Bent Panels			Rounds	
Steel Coating Location Replicate	$\bar{X}$ Mils	Topside	Underside	$\bar{X}$ Mils	Topside	Underside	$\bar{X}$ Mils	Sides
Low-alloy steel I, II, III, and IV (codes 2, 3, 4, and 0 (no code)) —*Continued*								
2-7-12-17	3.68	TrR	0	2.52	10R, 5Y[h]	30R, 10Y[h]	...	...
2-7-12-18	...	...	...	2.60	TrY[e]	TrR, 5Y	...	...
3-7-12-14	...	...	...	2.49	5R[i]	10R, TrY[i]	...	N
3-7-12-15	...	...	...	2.45	5R[i]	10R, 5Y[i]	...	N
3-7-12-16	2.87	10R[h]	20R, TrY[h]	2.06	10R[i]	5R, TrY[i]	...	N
3-7-12-17	3.59	0	TrY[c]	1.90	5Y	5R, TrY[i]	...	N
3-7-12-18	3.14	0	TrY[c]	3.41	0	TrY	...	N
4-7-12-14	...	...	...	2.59	5R[i]	TrR, 5Y	...	...
4-7-12-15	...	...	...	2.80	0	TrY[c]	...	...
4-7-12-16	4.12	0	TrY[e]	2.39	5Y	5R, TrY[i]	...	...
4-7-12-17	3.99	0	TrY[e]	2.46	TrR, TrY	TrR	...	...
4-7-12-18	...	...	...	2.76	TrR	0	...	...
0-7-12-14	2.40	5R[i]	10R[g]	2.10	TrY[e]	0	1.06	25R[f,g]
0-7-12-15	...	...	...	2.45	0	TrY[e]	...	...
0-7-12-16	...	...	...	2.02	TrY	TrR	...	...
0-7-12-18	...	...	...	2.32	0	15R, 5Y[g]	...	...
Sprayed aluminum (code 10)								
Carbon steel (code 1)								
1-10-12-14	...	TrY[c]	0	...	TrY[c]	0	...	N
1-10-12-15	...	TrY[c]	0	...	TrY[c]	0	...	N
1-10-12-16	...	TrY[c]	0	...	TrY[c]	0	...	N
1-10-12-17	...	TrY[c]	0	...	TrY[c]	0	...	N
1-10-12-18	...	TrY[c]	0	...	TrY[c]	0	...	N
Low-alloy steel I and IV (codes 2 and 0 (no code))								
2-10-12-14	...	TrY[c]	0	...	TrY[c]	0	...	N
2-10-12-15	...	TrY[c]	0	...	TrY[c]	0	...	N
2-10-12-16	...	TrY[c]	0	...	TrY[c]	0	...	N
2-10-12-17	...	TrY[c]	0	...	TrY[c]	0	...	N
2-10-12-18	...	TrY[c]	0	...	TrY[c]	0	...	N
0-10-12-14	...	TrY[c]	0	...	TrY[c]	0	...	N
0-10-12-15	...	TrY[c]	0	...	TrY[c]	0	...	N
0-10-12-16	...	TrY[c]	0	...	TrY[c]	0	...	N
0-10-12-17	...	TrY[c]	0	...	TrY[c]	0	...	N
0-10-12-18	...	TrY[c]	0	...	TrY[c]	0	...	N

[a] Hot-dip zinc samples (code 5) were centrifuged immediately after coating to remove excess zinc. Details are contained in the 1959 ASTM *Proceedings*, p. 137.

[b] Mechanical damage on left edge. [c] Around hole(s).

[d] Hot-dip aluminum samples (code 6) were prepared by immersing in the aluminizing bath for 1 min; code 7 samples were immersed for 2 min and rinsed in the fluxing bath immediately after coating to remove excess aluminum. More complete details are found in the 1959 ASTM *Proceedings*, p. 137.

[e] At edge. [f] On end.

[g] 5R or more, first noted during inspection on 5-31-60.

[h] 5R or more, first noted during inspection on 5-23-61.

[i] 5R or more, first noted during inspection on 6-5-62.

[j] **Specimens not showing a trace of yellow or rust are not included, hence the blank spaces in columns.**

TABLE III.—REPORT OF INSPECTION OF COATED STEEL HARDWARE, KURE BEACH, N.C. (80-FT SITE, CODE 13).

Exposed: 6-23-58; inspected: 6-5-62; exposure: 3.94 years.

Abbreviations and symbols: 0 = no rust; 5, 10, 25, 100 = approximate percentage of rust; R = rusting of base metal; Y = yellow or orange rust; Tr = trace; N = no specimen exposed; $\bar{X}$ = average measured coating thickness.

Specimen	Flat Panels			Bent Panels			Rounds	
Steel Coating Location Replicate	$\bar{X}$ Mils	Topside	Underside	$\bar{X}$ Mils	Topside	Underside	$\bar{X}$ Mils	Sides
Hot-dip zinc (code 5)[a]								
Carbon steel (code 1)								
1-5-13-18	2.40	TrY[b]	0	...	...	...	...	...
Low-alloy Steel I and III (codes 2 and 4)								
2-5-13-15	...	...	...	...	...	...	5.50	TrY[d]
4-5-13-18	...	...	...	2.81	0	TrY[c]	...	...
Hot-dip aluminum (code 6)[e]								
Carbon steel (code 1)								
1-6-13-14	...	...	...	...	...	...	2.71	TrY[d]
1-6-13-17	...	...	...	...	...	...	2.86	TrY[d]
Low-alloy steel I, II, III, and IV (codes 2, 3, 4, and 0 (no code))								
2-6-13-14	...	...	...	1.74	TrY[f]	TrY	...	...
2-6-13-16	...	...	...	...	...	...	3.37	TrY[d]
3-6-13-14	3.31	TrY[b]	0	...	...	...	...	N
3-6-13-16	2.33	TrY[f]	TrY[f]	1.69	0	TrY	...	N
3-6-13-18	2.70	0	TrY	...	...	...	...	N
4-6-13-14	...	...	...	2.62	TrY	0	...	...
4-6-13-15	...	...	...	2.34	TrY	0	4.17	TrY
4-6-13-16	...	...	...	2.29	TrY	0	...	...
4-6-13-17	...	...	...	1.96	TrY[b]	0	...	...
4-6-13-18	2.43	TrY[b]	TrY[f]	...	...	...	...	...
0-6-13-15	2.77	TrY	0	...	...	...	...	...
0-6-13-18	2.58	TrY[b]	0	...	...	...	...	...
Hot-dip aluminum (code 7)[e]								
Carbon steel (code 1)								
1-7-13-14	2.28	10R[g]	TrR	...	...	...	...	...
Low-alloy steel I, II, III, and IV (codes 2, 3, 4, and 0 (no code))								
2-7-13-14	3.01	25R, 10Y[h]	50R, 25Y[h]	2.21	40R[h]	25R, 15Y[g]	...	...
2-7-13-15	...	...	...	3.30	5R[i]	0	...	...
2-7-13-16	...	...	...	2.30	10R[h]	10R, 10Y[g]	...	...
2-7-13-17	...	...	...	3.31	TrR	TrR, TrY	...	...
2-7-13-18	...	...	...	2.25	50R, 10Y[g]	5R, TrY[g]	...	...
3-7-13-14	...	....	...	2.26	10R, 5Y[h]	10R, 5Y[g]	...	N
3-7-13-15	...	...	...	2.35	5R, 5Y[i]	TrY	...	N
3-7-13-16	...	...	...	2.36	TrR, TrY	TrY	...	N
3-7-13-17	...	...	...	2.40	TrR, 10Y	5R, TrY[g]	...	N
3-7-13-18	2.84	TrR	0	2.16	TrR, 15Y	TrR, TrY	...	N
4-7-13-16	...	...	...	2.59	TrY	TrY	...	...

TABLE III.—REPORT OF INSPECTION OF COATED STEEL HARDWARE, KURE BEACH, N. C. (80-FT SITE, CODE 13)—*Continued.*

Specimen	Flat Panels			Bent Panels			Rounds	
Steel Coating Location Replicate	X̄ Mils	Topside	Underside	X̄ Mils	Topside	Underside	X̄ Mils	Sides
Low-alloy steel I, II, III, and IV (codes 2, 3, 4, and 0 (no code))—*Continued*								
4-7-13-17	...	...	...	...	...	...	4.40	TrY
4-7-13-18	...	...	...	...	...	...	4.04	TrY
0-7-13-14	3.53	5R[i]	0	1.69	25R[h]	10R, 10Y[g]	...	...
0-7-13-15	3.33	TrR	0	1.84	20R[h]	TrR, TrY	...	...
0-7-13-16	...	...	...	1.91	10R[i]	0	...	...
0-7-13-17	...	...	...	2.04	20R, 10Y[h]	10R, 10Y[g]	...	...
0-7-13-18	3.12	TrR	TrY	1.86	10R[i]	TrR, TrY	...	...
Electroplated zinc (code 8)								
Carbon steel (code 1)								
1-8-13-15	2.17	TrY[b]	0	2.41	TrY	0	...	...
1-8-13-16	1.68	TrY[b]	0	2.38	TrY	0	...	...
1-8-13-17	...	...	...	2.25	TrY	0	...	...
1-8-13-18	2.53	0	20R[h]	...	...	...	...	...
Low-alloy steel I (code 2)								
2-8-13-14	...	...	...	...	...	...	2.15	5R[i]
2-8-13-15	...	...	...	...	...	...	3.20	TrR
2-8-13-16	...	...	...	...	...	...	2.24	10R[g]
2-8-13-18	...	...	...	...	...	...	2.14	TrR

[a] Hot-dip zinc samples (code 5) were centrifuged immediately after coating to remove excess zinc. Details are contained in the 1959 ASTM *Proceedings*, p. 137.

[b] At edge.

[c] Around mechanical scratch.

[d] On end.

[e] Hot-dip aluminum (code 6) samples were prepared by immersing in the aluminizing bath for 1 min; code 7 samples were immersed for 2 min and rinsed in the fluxing bath immediately after coating, to remove excess aluminum. More complete details are found in the 1959 ASTM *Proceedings*, p. 137.

[f] Around hole(s).

[g] 5R or more, first noted during inspection on 5–23–61.

[h] 5R or more, first noted during inspection on 5–31–60.

[i] 5R or more, first noted during inspection on 6–5–62.

10) show a trace of yellow only around the identification holes.

A few hot-dip zinc specimens (code 5) show a trace of yellow but no evidence of rust. Only the electroplated zinc (code 8) and sprayed zinc (code 9) specimens show no trace of yellow or rust. See Table II for an itemized report.

80-ft Site:

Two types of coated specimens show 5 per cent R or more rust of the base metal. Of the hot-dip aluminum specimens (code 7), 3 are flat and 12 are bent, 5 more than were noted in 1961 or earlier. Of the electroplated zinc specimens (code 8), 1 is flat and 2 are round, 1 more than noted in 1961.

A few hot-dip zinc (code 5) and hot-dip aluminum (code 6) specimens show a trace of yellow but no evidence of rust. A few of the hot-dip zinc specimens show signs of dark discoloration, as much as 10 per cent on a surface (top or bottom). The condition is not as pronounced as at the Newark site. No conclusions have been drawn.

No sprayed zinc (code 9) and sprayed aluminum (code 10) specimens were exposed. See Table III, for an itemized report.

Weather conditions in 1962 at Kure Beach were more favorable than in 1961, and some specimens appear to have improved in appearance. Obviously this cannot be true so that slight differences between reports should be considered as of little consequence.

Respectfully submitted on behalf of the subcommittee,

L. E. FIORITO,
Chairman

REPORT OF COMMITTEE A-6
ON
MAGNETIC PROPERTIES*

Committee A-6 on Magnetic Properties held two general meetings during the year: on June 26, 1962, during the Annual Meeting of the Society in New York City, and on Feb. 6, 1963, during Committee Week in Montreal, Quebec. Subcommittee and task group meetings were held on the previous day in both cases.

Membership in the committee remains at 43 with no changes in representation, but with some changes in personnel. Of the 43 members, 20 are classified as producers, 15 as consumers and 8 as general interest members. There have been no changes in organization except for realignment of some of the task groups.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1962 Annual Meeting, Committee A-6 presented to the Society through the Administrative Committee on Standards, the recommendation that the Standard Methods of Testing Magnetic Materials (A 34–55) be revised and reverted to tentative. This recommendation was accepted by the Standards Committee on Sept. 11, 1962, and the revised Methods A 34–62 T appear in the 1962 Supplement to Book of ASTM Standards, Part 3.

RECOMMENDATIONS AT ANNUAL MEETING

Tentatives Continued Without Revision

The committee recommends the continuation without revision of the following tentatives:

Tentative Definitions of Terms, Symbols, and Conversion Factors Relating to Magnetic Testing (**A 340 – 61 T**) (being revised by the subcommittee).

Tentative Methods of Test for Permeability of Feebly Magnetic Materials (**A 342 – 60 T**) (revision out to letter ballot of the committee).

Tentative Methods of Test for Alternating Current Magnetic Properties of Materials Using Epstein Specimens (**A 343 – 60 T**) (revision of Sections 34 to 40 on high frequency is ready for letter ballot of the subcommittee).

Tentative Methods of Test for Electrical and Mechanical Properties of Magnetic Materials (**A 344 – 60 T**) (revision of Sections 4 to 18 on surface insulation resistance is ready for letter ballot of the subcommittee).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Methods of Sampling and Test (J. W. Hale, chairman) has been working on editorial changes in the Standard Methods of Test for Normal Induction and Hysteresis of Magnetic Materials (A 341) which

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include corrections and revision of text, figures, and symbols. Similar work has also been done on the Tentative Methods of Test for Permeability of Feebly Magnetic Materials (A 342).

Subcommittee IV on Material Specifications (A. C. Beiler, chairman) held two meetings during the year and is well on the way to drafts of new material specifications for the major commercial products. The subcommittee recommends that the Standard Specification for Flat-Rolled Electrical Steel (A 345) be continued without revision.

The Task Group on Electrical and Mechanical Properties drafted a revision of Sections 4 to 18 of the Tentative Methods of Test for Electrical and Mechanical Properties of Magnetic Materials (A 344) and is now engaged in restating portions of it which will be proposed prior to adoption of this method as standard.

The Task Group on Magnetic Shielding has been working on drafts of test methods for measurements of shielding materials and the Task Group on Magnetic Amplifier Core Materials is conducting additional tests of annealing procedures. Additional permanent magnet samples have been included in the round-robin tests of the Task Group on D-C Testing of High Coercive Materials.

This report has been submitted to letter ballot of the committee, which consists of 39 voting members; 28 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

A. C. BEILER,
Chairman.

W. S. EBERLY,
Secretary.

REPORT OF COMMITTEE A-10

ON

IRON-CHROMIUM, IRON-CHROMIUM-NICKEL, AND RELATED ALLOYS*

Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys and most of its subcommittees held two meetings during the year: in June, 1962, in connection with the Annual Meeting of the Society in New York, N. Y., and in Detroit, Mich., on Jan. 21 to 24, 1963, in conjunction with several other metals committees.

The committee has a total membership of 179, of whom 92 are classified as producers, 55 as consumers, and 32 as general interest members.

It is the expressed intent of Committee A-10 to avoid the use of tentatives, and all current tentatives not being revised will be adopted as standard. Additionally, these will then be recommended for approval as American Standard by the American Standards Assn.

Recommendations Accepted by the Administrative Committee on Standards

Subsequent to the 1962 Annual Meeting, Committee A-10 presented to the Society through the Administrative Committee on Standards the recommendations listed below. They were accepted by the administrative committee on the dates indicated. The new Tentative Specification A 484 is available as a separate reprint; the other recommendations appear in the 1962 Supplement to Book of ASTM Standards, Part 1.

New Tentative Specifications for:

Stainless and Heat Resisting Steel Forgings (A 473 – 62 T) (Accepted July 27, 1962),

Hot Worked, Hot-Cold Worked and Cold Worked Alloy Forgings and Forging Billets for High Strength at Elevated Temperatures (A 477 – 62 T) (Accepted Oct. 24, 1962),

Chromium-Nickel Stainless Steel Weaving Wire (A 478 – 62 T) (Accepted Oct. 24, 1962), and

Stainless and Heat Resisting Steel Bars and Shapes for Use in Boilers and Other Pressure Vessels (A 479 – 62 T) (Accepted Oct. 24, 1962).

New Tentative General Requirement Specifications for:

Delivery of Flat Rolled Stainless and Heat Resisting Steels in Form of Plate, Sheet, and Strip (A 480 – 62 T) (Accepted Oct. 17, 1962), and

Stainless and Heat Resisting Wrought Steel Products (Except Wire) (A 484 – 63 T) (Accepted March 21, 1963).

Revision and Reversion to Tentative of Standard Specifications for: (Two separate revisions—accepted July 23 and Sept. 11, 1962):

Seamless and Welded Ferritic Stainless Steel Tubing for General Service (A 268 – 62),

Seamless and Welded Austenitic Stainless Steel Tubing for General Service (A 269 – 62),

Seamless and Welded Austenitic Stainless Steel Sanitary Tubing (A 270 – 62), and

Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 – 61).

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RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee A-10 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] All of the existing tentatives and standards referred to appear in their present form in the 1961 Book of ASTM Standards, Part 1, or the 1962 Supplement thereto.

New Standards

as appended hereto[2]
(require nine-tenths affirmative vote)

Standard Specifications for:

Stainless and Heat-Resisting Steel Rope Wire, and
Stainless and Heat-Resisting Steel Cold Heading Wire.

Revision of Tentatives

Tentative Specification for Corrosion-Resistant Iron-Chromium and Iron-Chromium-Nickel Alloy Castings for General Application (A 296 - 62 T):

Table II.—Change Footnote *d* to read as follows:

[d] Grade CF-8C shall have a columbium content of not less than 8 times the carbon content and not more than 1.0 per cent. If a columbium-plus-tantalum alloy in the approximate Cb:Ta ratio of 3:1 is used for stabilizing this grade, the total columbium-plus-tantalum content shall not be less than nine times the carbon content and shall not exceed 1.1 per cent.

Tentative Specification for Seamless and Welded Austenitic Stainless Steel Pipe (A 312 - 62 T):

New Section 3.—Add a new Section 3 entitled "Basis of Purchase" to read as follows, renumbering the present section accordingly:

3. Orders for material under this specification shall include the following, as required, to describe the desired material adequately:

(*1*) Quantity (feet or number of lengths),
(*2*) Name of material (austenitic steel pipe),
(*3*) Process (seamless or welded),
(*4*) Grade (Table I),
(*5*) Size (nominal size or outside diameter and schedule number or average wall thickness),
(*6*) Length (specific or random, Section 17),
(*7*) End finish (Section 18),
(*8*) Optional requirements (check analysis, Section 8; supplementary requirements S1 to S5),
(*9*) Test report required (Section 21(*b*)),
(*10*) ASTM designation, and
(*11*) Special requirements or exception to the specification.

Section 11(a).—Change definitions of "t" and "D" to read as follows:

t = specified wall thickness, in inches, and
D = specified outside diameter, in inches.

Tentative Specification for Centrifugally Cast Austenitic Steel Pipe for High-Temperature Service (A 451 - 62 T):

New Section 3.—Add a new Section 3 entitled "Basis of Purchase" to read as follows, renumbering the present sections accordingly:

3. Orders for material under this specification shall include the following, as required, to describe the desired material adequately:

(*1*) Quantity (feet or number of lengths),
(*2*) Name of material (centrifugally cast pipe),
(*3*) Grade (Table II),
(*4*) Size (outside or inside diameter and minimum wall thickness),
(*5*) Length (specific or random, Section 16),
(*6*) End finish (Section 17),
(*7*) Optional requirements (check analysis, Section 7; special hydrostatic test, Section 10, Note 1; repair by welding, Section 18 (*c*); supplementary requirements S1 to S7),
(*8*) Test report required (Section 20(*a*)),
(*9*) ASTM designation, and
(*10*) Special requirements or exceptions to specification.

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[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[2] The new standards appear in the *1964 Book of ASTM Standards*, Part 3.

Section 10(a).—Change definitions of "*t*" and "*D*" as indicated above for Section 11(*a*) of Specification A 312.

Tentative Specification for Centrifugally Cast Austenitic Cold-Wrought Pipe for High-Temperature Service (A 452 - 62 T):

New Section 3.—Add a new Section 3 entitled "Basis of Purchase" to read as follows, renumbering the present sections accordingly:

3. Orders for material under this specification shall include the following, as required, to describe the desired material adequately:

(*1*) Quantity (feet or number of lengths),

(*2*) Name of material (centrifugally cast cold-wrought pipe),

(*3*) Grade (Table I),

(*4*) Size (inside diameter and minimum wall thickness),

(*5*) Length (specific or random, Section 17),

(*6*) End finish (Section 18),

(*7*) Optional requirements (check analysis, Section 8; special hydrostatic test, Section 11, Note 2; repair by welding, Section 19(*c*); supplementary requirements S1 to S7),

(*8*) Test report required (Section 21(*c*)),

(*9*) ASTM Designation, and

(*10*) Special requirements or exceptions to specification.

TABLE II.—ABRASIVE CUTTING WIDTH AND LENGTH TOLERANCES FOR STAINLESS AND HEAT RESISTING STEELS.

(New Table XIX for Specification A 240)

Specified Thickness, in.	Tolerance Over Specified Width and Length, in.[a] [b]	
	Width	Length
Up to 1¼, incl.	⅛	⅛
Over 1¼ to 2¾, incl.	3/16	3/16

[a] The tolerance under specified width and length is ⅛ in.

[b] Width and length tolerances for abrasive cut plates over 2¾ in. thick are not included.

Section 12(a).—Change definitions of "*t*" and "*D*" as indicated above for Section 11(*a*) of Specification A 312.

Add a new Section 3 on "Basis of Purchase" similar to those indicated above for Specifications A 312, A 451, and A 452 to the following specifications:

Tentative Specifications for:

Seamless and Welded Ferritic Stainless Steel Tubing for General Service (**A 268 - 62 T**),
Seamless and Welded Austenitic Stainless Steel Tubing for General Service (**A 269 - 62 T**),
Seamless and Welded Austenitic Stainless Steel Sanitary Tubing (**A 270 - 62 T**), and
Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (**A 271 - 62 T**).

Adoption of Tentatives as Standard Without Revision

Tentative Specifications for:

Corrosion-Resisting Chromium-Nickel Steel Plate, Sheet, and Strip (**A 167 - 61 T**),
Corrosion-Resisting Chromium Steel Plate, Sheet, and Strip (**A 176 - 61 T**),
Corrosion-Resisting Chromium Steel Clad Plate, Sheet, and Strip (**A 263 - 61 T**),
Corrosion-Resisting Chromium-Nickel Steel Clad Plate, Sheet, and Strip (**A 264 - 61 T**),
Heat-Resistant Iron-Chromium and Iron-Chromium-Nickel Alloy Castings for General Application (**A 297 - 60 T**),
Iron-Chromium and Iron-Chromium-Nickel Alloy Tubular Centrifugal Castings for General Applications (**A 362 - 52 T**),
High-Strength, High-Temperature Bolting Materials with Expansion Coefficients Comparable to Austenitic Steels (**A 453 - 61 T**),
Hot, Hot-Cold-Worked, and Cold-Worked Alloy Steel Plate, Sheet, and Strip for High Strength at Elevated Temperatures (**A 457 - 61 T**),
Hot, Hot-Cold-Worked, and Cold-Worked Alloy Steel Bars for High Strength at Elevated Temperatures (**A 458 - 61 T**), and
General Requirements for Delivery of Flat Rolled Stainless and Heat Resisting Steels in Form of Plate, Sheet, and Strip (**A 480 - 62 T**).

Tentative Method of:

Total Immersion Corrosion Test of Stainless Steels (**A 279 - 44 T**).

Tentative Recommended Practices for:

Boiling Nitric Acid Test for Corrosion-Resisting Steels (**A 262 - 55 T**), and
Conducting Acidified Copper Sulfate Test for Intergranular Attack in Austenitic Stainless Steel (**A 393 - 55 T**).

Adoption of Tentatives as Standard With Revisions

Tentative Specification for Corrosion-Resisting Chromium and Chromium-Nickel Steel Plate, Sheet, and Strip for Fusion-Welded Unfired Pressure Vessels (A 240 - 61 T):

Table XIII.—Change to read as shown in the accompanying Table I.[3]

Table XVIII.—Change to read as follows and add to the title the words "Stainless and Heat Resisting Steels":

Specified Thickness, in.	Machining Allowance per Edge, in.
2 and under	1/4
Over 2 to 3, incl	3/8
Over 3 to 6, incl.[a]	1/2

[a] Cutting allowances for plates over 6 in. thick are not included herein.

New Table XIX.—Add a new Table XIX to read as shown in the accompanying Table II.

Tentative Specification for Corrosion-Resisting Chromium-Nickel-Manganese Steel Plate, Sheet, and Strip (A 412 - 57 T):

Revise as appended hereto.[4]

Tentative Specifications for Hot-Rolled and Cold-Finished Corrosion-Resisting Chromium - Nickel - Manganese Steel Bars (A 429 - 58 T):

Section 14.—Change to read as follows:

14. The inspector representing the purchaser shall have access to the place where the material for acceptance is kept and shall have the right to witness the selection of samples, the preparation of the test pieces, and the performance of the test, or tests. The inspector shall have the right to indicate the pieces from which the samples will be selected in accordance with the provisions of this specification.

Section 16.—Change the rejection period to read "60 working days from the receipt of the material by the purchaser."

Tentative Specification for Stainless and Heat Resisting Steel Forgings (A 473 - 62 T):

Sections 16(a) and 17.—Change as indicated above for Sections 14 and 16 of Specification A 429.

Tentative Specification for Hot Worked, Hot-Cold Worked and Cold Worked Alloy Steel Forgings and Forging Billets for High Strength at Elevated Temperatures (A 477 - 62 T):

Sections 16 and 17(a).—Change as indicated above for Sections 14 and 16 of Specification A 429.

Tentative Specification for Chromium-Nickel Stainless Steel Weaving Wire (A 478 - 62 T):

Sections 14 and 15(a).—Change as indicated above for Sections 14 and 16 of Specification A 429.

Tentative Specification for Stainless and Heat-Resisting Steel Bars and Shapes for Use in Boilers and Other Pressure Vessels (A 479 - 62 T):

Sections 18(a) and 19(a).—Change as indicated above for Sections 14 and 16 of Specification A 429.

Revision of Standards, Immediate Adoption

(requires nine-tenths affirmative vote)

Standard Specification for Hot-Rolled and Cold-Finished Stainless and Heat Resisting Steel Bars (A 276 - 62):

Sections 14 and 16.—Change as indicated above for Sections 14 and 16 of Specification A 429.

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[3] Table I is not included in this report but appears in the revised specification; see *1964 Book of ASTM Standards*, Part 3.

[4] The revised standard appears in the *1964 Book of ASTM Standards*, Part 3.

Standard Specifications for Chromium-Nickel Corrosion-Resisting Steel Spring Wire (A 313 – 55):

Sections 13 and 14.—Change as indicated above for Sections 14 and 16 of Specification A 429.

Standard Specifications for Corrosion Resisting Steel Billets and Bars for Reforging (A 314 – 58):

Sections 11 and 12.—Change as indicated above for Sections 14 and 16 of Specification A 429.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II, Editorial, (A. G. Cook, chairman) has been concerned with the editorial review of the new or revised specifications. This was done to assure that these specifications had the editorial uniformity recommended by Committee A-10 in the formation of Subcommittee II.

The Recommended Editorial Procedure and Form, and the Glossary of Recommended Abbreviations and Forms were distributed to the members of Committee A-10 and the interested members of the other committees of the Society.

In collaboration with several other metals committees, a task group is preparing specific recommendations for the use of the point system of numbering paragraphs to be submitted to the Society.

Subcommittee IV on Methods of Corrosion Testing (O. B. Ellis, chairman) has completed the task group work investigating methods of detecting susceptibility to intergranular corrosion in austenitic stainless steels. The task group has prepared the first drafts of revisions in the oxalic acid etching test and the boiling nitric acid test as now described in the Recommended Practice for Boiling Nitric Acid Test for Corrosion-Resisting Steels (A 262). Also, first drafts of the ferric sulfate-sulfuric acid test for detecting susceptibility to intergranular attack in stainless steels and the 10 per cent nitric-3 per cent hydrofluoric acid test for detecting susceptibility to intergranular attack in molybdenum-bearing stainless steels have been prepared. These four procedures will be combined into a single recommended practice for submission to members of Committee A-10.

Work will be done to study the acceleration of the acidified copper sulfate test (A 393) by addition of copper chips to the test solution. It is planned to combine this test with the four described above so that all methods for detecting susceptibility to intergranular attack in austenitic stainless steels will be in one recommended practice.

The Task Group on Atmospheric Corrosion Tests has not received all reports on mechanical properties of welded specimens exposed at six test sites for three years. The data obtained on unwelded specimens indicate no significant change in mechanical properties of specimens exposed at State College, Pa., Newark, N. J., Columbus, Ohio, Brazos River (Freeport), Tex., and the 80 ft and 800 ft lots at Kure Beach, N. C.

Subcommittee V on Mechanical Testing (R. H. Heyer, chairman).—A task group on hardness conversions is conducting interlaboratory tests on test blocks of austenitic stainless grades for Rockwell, superficial Rockwell, and diamond pyramid hardness tests. Out of eleven laboratories six have already reported the data, and it is expected that the analysis will be completed and the conversion relationships will be available at the June meeting.

The task group on tension testing of thin gage flat-rolled stainless steel has drafted a method of testing these materials, based on results for comparative tests, and is planning a second series of tests to check out the method.

Subcommittee VI on Metallography (G. A. Sands, chairman) has prepared samples of type 316 stainless steel for studies of the microstructure. This includes the customary light-microscope studies, and has been expanded in cooperation with Subcommittee XI of Committee E-4 on Metallography to embrace thin foil studies and electron probe analysis.

Subcommittee VIII on Specifications for Wrought Products (R. B. Gunia, chairman) has been quite active during the past year. Specifications for Stainless Steel Bars and Shapes for Boilers and Pressure Vessels (A 479), for Stainless Steel Rope Wire, and for Stainless Steel Cold Heading Wire; and a General Requirement Specification for Delivery of Stainless Steel Wrought products were written and approved, as noted earlier in this report.

All tentatives were adopted as standard, and all future specifications will be prepared as standards.

Subcommittee IX for Flat Products (G. W. Hinkle, chairman) has approved some title changes to attain uniformity and revised Specifications A 240 and A 412. In addition, committee approval was given on the following items: (1) Adoption of all tentative specifications as standard, (2) A new write-up on plant inspection to be prepared by the editorial committee for all specifications and submitted to letter ballot, (3) Rejection paragraph changing the time limit to 60 days, and (4) Addition of metric system to all tables and properties in all specifications. This will be deferred until the procedure is clarified by ASTM Headquarters.

Subcommittee X on Specifications for Castings (H. C. Templeton, chairman) has approved and recommended the adoption of some heat treatment and mechanical property changes in several grades of alloys in Specification A 296.

One task group is currently engaged in a revision of Specification A 362, and another group has a draft proposal for a new specification covering high-silicon irons.

Subcommittee XI on Specifications for Tubular Products (J. J. B. Rutherford, chairman) has approved a task group report concerning hot ductility and ultrasonic tests for inclusion in Specifications A 376 and A 430. Another task group on nuclear requirements has approved recommendations for several specifications. In both instances, the recommendations have been submitted for approval in Committee A-10 and in Committee A-1 on Steel.

In accordance with Boiler Code comments, revisions have been made in the centrifugally cast pipe Specifications A 451 and A 452; these are being submitted for letter ballot in both Committees A-1 and A-10.

Subcommittee XII on Specifications for High Temperature, Super-Strength Alloys (W. F. Simmons, chairman) has completed a Specification for Precipitation Hardening Alloy Bars, Forgings, and Forging Stock. This has been submitted to the Standards Committee for approval.

Other task groups are carrying out assignments for: (1) Addition of 12 precipitation hardening alloys to the present specification at the request of the American Iron and Steel Inst. (AISI), (2) Conclusion of work on a draft covering 14 high-carbon, chromium-nickel casting alloys, and (3) Specifications for martensitic type high-temperature alloys at the request of AISI.

Subcommittee XIII on Specifications for Nuclear Reactor Structural Materials (C. R. Sutton, chairman) has approved of a task group recommendation that

Committee A-10 prepare a general purpose specification for materials for nuclear service. This would entail intersubcommittee effort and was remanded to the Advisory Committee for action.

This report has been submitted to letter ballot of the committee, which consists of 179 members; 115 members returned their ballots, of whom 104 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

L. L. WYMAN,
Chairman.

E. E. DENHARD,
Secretary.

REPORT OF COMMITTEE B-1

ON

WIRES FOR ELECTRICAL CONDUCTORS*

Committee B-1 on Wires for Electrical Conductors held three meetings during the year: in Niagara Falls, Ontario, on Sept. 18, 1962; in New York, N. Y., on Dec. 18, 1962; and in Williamsburg, Va., on March 5, 1963. Meetings of Subcommittees IV, V, VI, and VII also were held during the year.

The committee notes with regret, the retirement during the year of the following long-time members who have contributed much to the committee's work: J. L. Thomas, L. B. Curtis, and C. E. Topping. During the year, M. V. Yokelson resigned as chairman of Subcommittee IV due to pressure of other work. The Advisory Subcommittee took action to designate Robert A. Harris to fill the unexpired term of L. B. Curtis as Member-at-Large of the Advisory Subcommittee, D. H. Sandell to fill the unexpired term of Mr. Topping as vice-chairman of Committee B-1, and H. M. Specht to fill the unexpired term of Mr. Yokelson as chairman of Subcommittee IV.

The committee consists of 81 members, of whom 41 are classified as producers, 26 as consumers, and 14 as general interest members.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1962 Annual Meeting, Committee B-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for:

Concentric - Lay - Stranded Aluminum Conductors, Steel Reinforced (ACSR) (B 232 – 60 T),

Aluminum-Coated (Aluminized) Steel Core Wire for Aluminum Conductors, Steel Reinforced (ACSR) (B 341 – 60 T).

Nickel-Coated Soft or Annealed Copper Wire (B 355 - 60 T).

Revision of Tentative Definitions for:

Terms Relating to Uninsulated Metallic Electrical Conductors (B 354 – 61 T).

Tentative Continued Without Revision:

Specifications for Copper Conductors for Use in Hookup Wire for Electronic Equipment (B 286 – 60 T).

These recommendations were accepted by the Standards Committee on October 1, 1962, and the revised specifications and definitions appear in the 1962 Supplement to Book of ASTM Standards, Part 2. The Tentative Specification continued without revision appears in the 1961 Book of ASTM Standards, Part 2.

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee B-1 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] All of the existing tentatives and standards referred to in this report appear in their present form in the 1961 Book of ASTM Standards, Part 2, or in the 1962 Supplement.

New Tentatives

During the past few years there has been growing commercial interest in aluminum-alloy conductors, aluminum-clad steel wire for use as conductors or as mechanical reinforcement in ACSR, and compacted aluminum and ACSR conductors. About three years ago it was reported to the committee that a number of utility members of the Edison Electric Institute had indicated interest in these constructions as transmission and distribution conductors or as neutrals for service drop cables. Accordingly, as part of its continuing program of developing proposed specifications for several particular types of aluminum conductors, the committee started developing suitable specification requirements for these types of conductors. The following 8 proposed specifications as appended hereto,[2] are recommended to supplement the present specifications for aluminum and ACSR conductors:

New Tentative Specifications for:

Aluminum Alloy 5005-H19 Wire for Electrical Purposes,
Concentric-Lay-Stranded Aluminum Alloy 5005-H19 Conductors,
Aluminum Alloy 6201-T81 Wire for Electrical Purposes,
Concentric-Lay-Stranded Aluminum Alloy 6201-T81 Conductors,
Hard-Drawn Aluminum-Clad Steel Wire,[3]
Concentric-Lay-Stranded Aluminum-Clad Steel Conductors,[3]
Compact Round Concentric-Lay-Stranded EC Grade Aluminum Conductors, Hard-Drawn, and
Compact Round Concentric-Lay-Stranded Aluminum Conductors, Steel-Reinforced (ACSR).

Revision of Tentatives

Tentative Specification for Concentric-Lay-Stranded Aluminum Conductors, Steel-Reinforced (ACSR) (B 232 - 62 T):

New Section 12.—Insert a new Section 12 entitled "Finish" to read as follows renumbering subsequent sections accordingly: "12. The conductor shall be free from all imperfections not consistent with the best commercial practice."

Section 12(a).—Renumber as Section 13(*a*) and add a second sentence to read: "Recommended package sizes are shown in Table V."

Table V.—Change to read as shown in the accompanying Table I, retaining present note.[4]

Table VI.—Add a new Table VI to read as shown in the accompanying Table II.[4]

Explanatory Note 2.—Change to read: "For definitions of terms relating to conductors, reference should be made to (*1*) American Standard Definitions of Electrical Terms (ASA No. C42.35—1957) and (*2*) Terms Relating to Uninsulated Metallic Electrical Conductors (ASTM Designation: B 354)."

Explanatory Note 6.—Change the last sentence to read: "It is also common practice to allow an amount not exceeding 10 per cent of the total weight of any one order to be shipped in random lengths but no piece shorter than 50 per cent of the standard length ordered."

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[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[2] The new specifications accepted at the Annual Meeting appear in the 1963 Supplement to *Book of ASTM Standards*, Part 2.

[3] These two specifications failed of approval at the Annual Meeting.—Ed.

[4] Tables I to VIII and Fig. 1 are not included in this report but appear in the revised tentatives; see 1963 Supplement to *Book of ASTM Standards*, Part 2.

Tentative Specification for Aluminum-Coated (Aluminized) Steel Core Wire for Aluminum Conductors, Steel Reinforced (ACSR) (B 341 – 62 T):

Section 3(a).—Change the beginning of this section to read: "The base metal shall be steel produced by either the acid or basic open-hearth, electric-furnace, or basic oxygen process and shall . . ."

Section 13(b).—Change to read "The length tolerances for the nominal coil lengths in Table V shall be plus and minus 2 per cent."

Section 14.—After "type of wire" add the words "nominal length of coil."

Table V.—Change to read as shown in the accompanying Table III.[4]

Tentative Definitions of Terms Relating to Uninsulated Metallic Electrical Conductors (B 354 – 62 T):

Introduction.—Add a new section entitled "Introduction" at the beginning of the definitions to read: "These definitions relate only to interpretations as applied to uninsulated metallic electrical conductors in specifications under the jurisdiction of ASTM Committee B-1 on Wires for Electrical Conductors, and do not necessarily correspond to the definitions used in other fields."

New Definitions.—Add the following 4 new definitions in alphabetical order:

Welding.—Joining the ends of two wires, rods, or groups of wires (*1*) by fusing, using the application of heat, pressure or both, by means of a flame torch, electric arc, or electric current or (*2*) by cold pressure.

ETP.—High conductivity, electrolytic tough pitch copper. Electrolytically refined copper cast so as to contain a controlled amount of oxygen for the purpose of obtaining a level set in the casting.

Stiffness.—(As applied to copper). The property of a conductor which causes it to resist permanent deformation by bending.

Intermediate Temper.—(As applied to aluminum). Any temper between soft and hard drawn.

Tentative Specification for Nickel-Coated Soft or Annealed Copper Wire (B 355 – 62 T):

Section 6(e)(2).—In the paragraph entitled "*Examination of Specimens,*" delete the word "peripheral" and change the word "parting" to "flaking."

Table II.—Change the resistivity value for Class 27 to read "1232.7" instead of "1232.1."

Table VI.—Change to read as shown in the accompanying Table IV.[4]

Adoption of Tentative as Standard Without Revision

Tentative Method of Test for Electrical Conductivity by Use of Eddy Currents (**B 342 – 61 T**).

Revision of Standards, Immediate Adoption

(requires nine-tenths affirmative vote)

Standard Specifications for Soft or Annealed Copper Wire (B 3 – 56):

Title.—Change "Specifications" to singular.

Section 1.—Change to start "This specification covers . . ."

Section 2.—Change to start "Orders for material under this specification shall . . ."

Section 3(a).—At the end of the first sentence, change "these specifications" to read "this specification."

Section 3(b).—Change "these specifications" to read "this specification.

Section 4(a).—Change to read as follows:

Tensile Strength and Elongation.—The wire shall conform to the requirements for elongation prescribed in Table I (Note 2). No requirements for tensile strength are specified. For wire whose nominal diameter is more than 0.001 in. (1 mil) greater than a size listed in Table I, but less than that of the next larger size, the requirements of the next larger size shall apply.

Table I.—Delete in its entirety the column headed "Tensile Strength, max, psi."

Section 5(c)(1).—Change to start "For elongation and resistivity determinations, the sample shall . . ."

Section 6(a).—Delete the first paragraph of this section, and replace it with: "No test for tensile strength shall be required."

Section 7.—Delete Paragraph (a) and reletter the ensuing sections in the following order: (*a*) *Elongation;* (*b*) *Resistivity;* (*c*) *Dimensions;* (*d*) *Surface Finish; and* (*e*) *Packaging.*

Standard Specifications for Bronze Trolley Wire (B 9 – 55):

Title.—Change "Specifications" to singular.

Section 1(a).—Change to start "This specification covers . . ."

Section 2.—Change to start "Orders for material under this specification shall . . ."

Section 3.—Change "these specifications" to read "this specification".

Figure 2.—Change the cross-section drawing of Figure-9 deep-section grooved trolley wire as shown in the accompanying Fig. 1.[4]

Under the cross-section drawing, add a note to read "Dimension H is defined by two center lines of which the upper is the center line of the radius of the groove and the lower is the center line of the groove."

In the table to the right of the figure, add the following in proper sequence:

A (at dimension N)	0.482(+0.006, −0.012)
F	0.250
H	0.005
P—Radius	0.037(±0.007)

Standard Specifications for Tinned Soft or Annealed Copper Wire for Electrical Purposes (B 33 – 58):

Title.—Change "Specifications" to singular.

Section 1.—Change to start "This specification covers . . ."

Section 2.—Change to start "Orders for material under this specification shall . . ."

Section 3(a).—Add a second paragraph to this section to read:

For purposes of this specification, the tin shall be considered "commercially pure" if the total of other elements, exclusive of copper, does not exceed 1 per cent. Notwithstanding the previous sentence, chemical analysis of the tin coating or of the tin used for coating shall not be required under this specification. Adequacy of the tin coating is assured by the continuity of coating and adherence of coating requirements (Section 4(*d*) and 4(*e*), respectively).

Section 3(b).—At the end of the first sentence, change "these specifications" to read "this specification."

Section 4(a).—Change to read as follows:

Tensile Strength and Elongation (Notes 2 and 3).—The tinned wire shall conform to the requirements for elongation prescribed in Table I. No requirements for tensile strength are specified. For wire whose nominal diameter is more than 0.001 in. (1 mil) greater than a size listed in Table I, but less than that of the next larger size, the requirements of the next larger size shall apply.

Table I.—Delete in its entirety the column headed "Tensile Strength, max, psi." Also, change the elongation requirement for 0.0113 diameter to read 15 instead of 10.

Section 5(c)(1).—Change to start "For elongation and resistivity determinations, the sample shall . . ."

Section 6(a).—Delete the first paragraph of this section, and replace it with: "No test for tensile strength shall be required."

Section 7.—Delete Paragraph (*a*) and reletter the ensuing sections in the following order: (*a*) *Elongation;* (*b*) *Resistivity;* (*c*) *Dimensions;* (*d*) *Continuity of Coating;* (*e*) *Adherence of Coating;* (*f*) *Surface Finish;* and (*g*) *Packaging.*

Standard Specifications for Soft Rectangular and Square Bare Copper Wire for Electrical Conductors (B 48 – 58):

Title.—Change "Specifications" to singular.

Section 1.—Change Paragraph (*a*) to start "This specification covers . . ."

Change Paragraph (*b*) to start "For the purpose of this specification . . ."

Section 2.—Change to start "Orders for material under this specification shall . . ."

Section 3.—Change to read identically with Section 3(*a*) and (*b*) of Specification B 3 (as revised in this report).

Section 5.—Change to start "For the purpose of this specification . . ."

Section 7.—Change Paragraph (*a*) to read "Type A wire shall conform to the following requirements for elongation. No requirements for tensile strength are specified."

In the tabular material included in this section, delete in its entirety the column headed "Tensile Strength, max, psi."

In Paragraph (*b*) delete the word "the" before "tensile."

In Paragraph (*c*) change the first word to "Elongation" instead of "Tension."

Standard Specifications for Lead-Coated and Lead-Alloy-Coated Soft Copper Wire for Electrical Purposes (B 189 – 58):

Title.—Change "Specifications" to singular.

Section 1.—Change to start "This specification covers . . ."

Section 2.—Change to start "Orders for material under this specification shall . . ."

Section 3(b).—At the end of the first sentence, change "these specifications" to read "this specification."

Section 4(a).—Change to read as follows:

> *Tensile Strength and Elongation* (Note 1).—The coated wire shall conform to the requirements for elongation prescribed in Table I. No requirements for tensile strength are specified. For wire whose nominal diameter is more than 0.001 in. (1 mil) greater than a size listed in Table I, but less than that of the next larger size, the requirements of the next larger size shall apply.

Table I.—Delete in its entirety the column headed "Tensile Strength, max, psi." Also, change the elongation requirement for 0.0113 diameter to read "15" instead of "10."

Section 5(c)(1).—Change to start "For elongation and resistivity determinations, the sample shall . . ."

Section 6(a).—Delete the first paragraph of this section, and replace with "No test for tensile strength shall be required."

Section 7.—Delete Paragraph (*a*) and reletter the ensuing sections in the following order: (*a*) *Elongation;* (*b*) *Resistivity;* (*c*) *Dimensions;* (*d*) *Continuity of Coating;* (*e*) *Surface Finish;* and (*f*) *Packaging.*

Standard Specifications for Concentric-Lay-Stranded Aluminum Conductors, Hard, Three-Quarter Hard, and Half-Hard (B 231 – 60):

Title.—Change "Specifications" to singular.

Section 1.—Change Paragraph (*a*) to start "This specification covers . . ."

Change Paragraph (*b*) to start "For the purpose of this specification . . ."

Section 2.—Change to start "Orders

for material under this specification shall . . ."

Change Paragraph (*2*) to read "Conductor size: circular-mil area or Awg (Section 6 and Tables II and III)."

Change Paragraph (*7*) to read "Package size (Section 12(*a*) and Table II)."

Section 6.—At end of this section, change "Table II" to read "Tables II and III."

Section 11.—At the end of the first sentence, change "Table II" to read "Table II or III."

New Section 12.—Insert a new Section 12 entitled "Finish" to read as follows renumbering subsequent sections accordingly: "12. The conductor shall be free from all imperfections not consistent with the best commercial practice."

Section 12(*a*).—Renumber as Section 13(*a*) and add a second sentence to read: "The recommended package sizes for Classes AA and A are shown in Table II."

New Table II.—Add a new Table II to read as shown in the accompanying Table V.[4]

Table II.—Change to read "Table III.—Construction Requirements of Concentric - Lay - Stranded Aluminum Conductors, Classes B, C, and D." Delete columns for Classes AA and A.

Table III.—Renumber as Table IV.

New Table V.—Add a new Table V to read as shown in the accompanying Table VI.[4]

Explanatory Note 2.—Change to read: "For definitions of terms relating to conductors, reference should be made to (*1*) American Standard Definitions of Electrical Terms (ASA No. C42.35—1957) and (*2*) Terms Relating to Uninsulated Metallic Electrical Conductors (ASTM Designation: B 354)."

Standard Specification for Standard Weight Zinc-Coated (Galvanized) Steel Core Wire for Aluminum Conductors, Steel Reinforced (ACSR) (B 245 – 62):

Section 3(*a*).—Change the beginning of this section to read "The base metal shall be steel produced by either the acid or basic open-hearth, electric-furnace, or basic oxygen process and shall . . ."

Section 13(*b*).—Change to read "The length tolerances for the nominal coil lengths in Table VI shall be plus and minus 2 per cent."

Section 14.—After "type of wire" add the words "nominal length of coil."

Table VI.—Change to read as shown in the accompanying Table VII.[4]

Standard Specification for Zinc-Coated (Galvanized) Steel Core Wire (With Coatings Heavier than Standard Weight) for Aluminum Conductors, Steel Reinforced (ACSR) (B 261 – 62):

Section 3(*a*).—Change the beginning of this section to read "The base metal shall be steel produced by either the acid or basic open-hearth, electric-furnace, or basic-oxygen process and shall . . ."

Table II.—Change initial setting for size 0.0500 to 0.0899 to read "0.0005" instead of "0.005."

Section 13(*b*).—Change to read "The length tolerances for the nominal coil lengths in Table VI shall be plus and minus 2 per cent."

Table VI.—Change to read as shown in the accompanying Table VIII.[4]

Standard Specifications for Silver-Coated Soft or Annealed Copper Wire (B 298 – 60):

Title.—Change "Specifications" to singular.

Section 1.—Change to start "This specification covers . . ."

Section 2.—Change to start "Orders for material under this specification shall . . ."

Section 3(a).—At the end of the first sentence, change "these specifications" to read "this specification."

Section 4(a).—Change to read as follows:

> 4.(*a*) *Tensile Properties.*—The silver-coated wire shall conform to the requirements for elongation prescribed in Table I. No requirements for tensile strength are specified. For wire whose nominal diameter is more than 0.001 in. (1 mil) greater than a size listed in Table I, but less than that of the next larger size, the requirements of the next larger size shall apply.

Table I.—Delete in its entirety the column headed "Tensile Strength, max, psi."

Section 5(c)(1).—Change to start "For elongation, resistivity, and weight of coating determinations, the sample shall . . ."

Section 6(a).—Delete the first paragraph of this section, and replace it with 'No test for tensile strength shall be required."

Section 7.—Delete Paragraph (*a*) and reletter the ensuing sections in the following order: (*a*) *Elongation;* (*b*) *Resistivity;* (*c*) *Dimensions;* (*d*) *Continuity of Coating;* (*e*) *Weight of Coating;* and (*f*) *Packaging.*

Editorial Changes

In the following specifications under the jurisdiction of Committee B-1, change the word "specifications" to read "specification" throughout: B 2, B 8, B 47, B 49, B 116, B 172, B 173, B 174, B 226, B 228, B 229, B 246.

Additional editorial changes are as follows:

Standard Specifications for Medium-Hard-Drawn Copper Wire (B 2 – 52):

Section 3.—Change to read identically with Section 3(*a*) and (*b*) of Specification B 3 (as revised in this report).

Standard Specifications for Copper Trolley Wire (B 47 – 55):

Section 3.—Change to read identically with Section 3(*a*) and (*b*) of Specification B 3 (as revised in this report).

Standard Specifications for Hot-Rolled Copper Rods for Electrical Purposes (B 49 – 52):

Section 3.—Change to read identically with Section 3(*a*) and (*b*) of Specification B 3 (as revised in this report).

Standard Specifications for Figure-9 Deep-Section Grooved and Figure-8 Copper Trolley Wire for Industrial Haulage (B 116 – 55):

Section 3.—Change to read identically with Section 3(*a*) and (*b*) of Specification B 3 (as revised in this report).

Standard Specifications for Tinned Hard-Drawn and Medium-Hard-Drawn Copper Wire for Electrical Purposes (B 246 – 58):

Section 2(b).—Revise to read identically with Section 3(*b*) of Specification B 33 (as revised in this report).

Standard Specifications for Concentric-Lay-Stranded Copper Conductors, Hard, Medium-Hard, or Soft (B 8 – 60):

Note 2.—Change to read "For definitions of terms relating to conductors, reference should be made to (*1*) American Standard Definitions of Electrical Terms (ASA No. C42.35—1957) and (*2*) Terms Relating to Uninsulated Metallic Electrical Conductors (ASTM Designation: B 354)."

Standard Specifications for Rope-Lay-Stranded Copper Conductors Having Bunch-Stranded Members, for Electrical Conductors (B 172 – 58):

Note 2.—Change to read "For definitions of terms relating to conductors, reference should be made to (*1*) American Standard Definitions of Electrical Terms (ASA No. C42.35—1957) and (*2*) Terms Relating to Uninsulated Metallic Electrical Conductors (ASTM Designation: B 354)."

Standard Specifications for Rope-Lay-Stranded Copper Conductors Having Concentric-Stranded Members, for Electrical Conductors (B 173 – 58):

Note 2.—Change to read "For definitions of terms relating to conductors, reference should be made to (*1*) American Standard Definitions of Electrical Terms (ASA No. C42.35—1957) and (*2*) Terms Relating to Uninsulated Metallic Electrical Conductors (ASTM Designation: B 354)."

Standard Specifications for Bunch-Stranded Copper Conductors for Electrical Conductors (B 174 – 59):

Note 2.—Change to read "For definitions of terms relating to conductors, reference should be made to (*1*) American Standard Definitions of Electrical Terms (ASA No. C42.35—1957) and (*2*) Terms Relating to Uninsulated Metallic Electrical Conductors (ASTM Designation: B 354)."

Standard Specifications for Cored, Annular, Concentric - Lay - Stranded Copper Conductors (B 226 – 56):

Note 2.—Change to read "For definitions of terms relating to conductors, reference should be made to (*1*) American Standard Definitions of Electrical Terms (ASA No. C42.35—1957) and (*2*) Terms Relating to Uninsulated Metallic Electrical Conductors (ASTM Designation: B 354)."

Standard Specifications for Concentric-Lay-Stranded Copper Covered Steel Conductors (B 228 – 56):

Note 2.—Change to read "For definitions of terms relating to conductors, reference should be made to (*1*) American Standard Definitions of Electrical Terms (ASA No. C42.35—1957) and (*2*) Terms Relating to Uninsulated Metallic Electrical Conductors (ASTM Designation: B 354)."

Standard Specifications for Concentric-Lay-Stranded Copper and Copper Covered Steel Composite Conductors (B 229 – 56):

Note 2.—Change to read "For definitions of terms relating to conductors, reference should be made to (*1*) American Standard Definitions of Electrical Terms (ASA No. C42.35—1957) and (*2*) Terms Relating to Uninsulated Metallic Electrical Conductors (ASTM Designation: B 354)."

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Editorial and Records (A. A. Jones, chairman) continued its editorial review of specifications and coordinated the various subcommittee recommendations regarding revisions of specifications.

Subcommittee II on Methods of Test and Sampling Procedures (R. H. Lloyd, chairman) recommended the adoption of Method B 342 as standard.

Subcommittee IV on Conductors of Copper and Copper Alloys (M. V. Yokelson, chairman [resigned–succeeded be H. M. Specht]) completed revisions of Specifications B 3, B 9, B 33, B 48, B 189, B 298 and B 355; and editorial revisions as noted earlier in the report.

The task group reviewing the need for solderability requirements reported that its contacts with the Electronic Industries Association (EIA) had indicated EIA sees no problem on solderability of wire except possibly for resistor leads and that both EIA and the Military have active groups working on this problem. Accordingly, the task group was discharged until such future time as any additional action might be required in ASTM.

Task groups are working on the preparation of proposed specifications for compact copper conductors (round, sector, segmental); reviewing the possibility (jointly with Committee B-2 on nonferrous metals and alloys) of developing a method of test for the annealing characteristic of copper; reviewing the requirements for electronic hookup wire; considering radius requirements of square and rectangular wire for film coating; considering requirements for bonded (heavy tin) hookup wire; and coordinating Awg standards in specification B 258 and National Bureau of Standards *Circular No. 31.* (*Circular No. 31* will no longer be published by NBS; accordingly, means are being studied for its publication by ASA or ASTM).

Subcommittee V on Conductors of Ferrous Metals (W. C. Clements, chairman) revised Specifications B 245, B 261, and B 341 as noted earlier in the report.

Task groups are studying the feasibility of permitting joints in finished galvanized or aluminized wires of stranded steel cores (jointly with Subcommittee VII); working on the preparation of proposed specifications for stranded steel cores for ACSR (jointly with Subcommittee VII); and considering the need for ductility requirements for various types of steel core wires (jointly with Subcommittees VI and VII).

Subcommittee VI on Composite Conductors (R. E. Koontz, chairman) prepared two new tentative specifications on aluminum-clad steel wire and strand as noted earlier in the report.

A task group is preparing a specification for copper-clad steel wire for electronic applications.

Subcommittee VII on Conductors of Light Metals (H. W. Adams, chairman) prepared four new specifications for aluminum alloy wire and strand, two new specifications for compacted aluminum and ACSR strand, and revised Specifications B 231 and B 232 as noted earlier in the report.

Task groups are reconsidering the inspection requirements in the various aluminum rod and wire specifications; considering the use of ASA standard temper designations in aluminum and aluminum alloy specifications; and reviewing the method of calculating rated strengths of aluminum-based conductors. Also, joint task groups are cooperating with Subcommittee V on three projects.

Joint Task Groups.—The joint task group on Glossary of Terms completed four new definitions and an introductory section for inclusion in Definitions B 354 as noted earlier in the report. It now is working on the preparation of additional definitions.

Over the past two years an attempt was made to establish a joint task group on Industry-Military specifications to coordinate military and industry requirements for essentially the same product, with particular regard to inspection and acceptance. Because of difficulty in obtaining suitable military representation, the proposal has been tabled.

A joint task group with Committee B-5 on Copper and Copper Alloys is making progress on revision of the resistivity requirements for DLP copper tubes in Specifications B 75 and B 372 (Wave Guide).

This report has been submitted to letter ballot of the committee, which consists of 81 members; 59½ members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

D. HALLORAN,
Chairman.

A. A. JONES,
Secretary.

REPORT OF COMMITTEE B-2
ON
NONFERROUS METALS AND ALLOYS*

Committee B-2 on Nonferrous Metals and Alloys held one meeting during the year on June 26, 1962, in New York, N. Y. Meetings of the Advisory Subcommittee were held on June 26, 1962, and Jan. 11, 1963, in New York, N. Y.

The committee consists of 144 members, of whom 65 are classified as producers, 56 as consumers, and 23 as general interest members.

Subcommittee V on Precious Metals and Alloys has been reactivated with C. D. Coxe as chairman and R. J. Lapee as secretary.

P. R. White replaced G. O. Hiers as chairman and D. M. Borcina was appointed secretary of Subcommittee III on Tin Base and Lead Base Alloys.

W. P. McFatridge replaced C. F. Crumbley as chairman of Subcommittee VII on Refined Nickel and Cobalt. Mr. Crumbley had earlier in the year replaced V. W. J. Franceschini in this post. G. A. Allison was appointed secretary.

The committee is considering incorporating in all specifications that refer to specific ASTM methods of analyses, a clause which would make mandatory the use of such ASTM methods only for referee purposes.

The committee has approved the transfer of the Specification for Nickel-Molybdenum and Nickel-Molybdenum-Chromium Alloy Castings (B 332) to the jurisdiction of Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys. Committee B-2 will retain jurisdiction over the wrought alloys that are presently under its Subcommittee VII and over wrought alloys in which nickel is the predominant ingredient.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1962 Annual Meeting, Committee B-2 presented to the Society through the Administrative Committee on Standards the following recommendations which were accepted by the Standards Committee on Oct. 17, 1962.

New Tentative Specifications for:

Columbium and Columbium Alloy Ingots (B 391 – 62 T),
Columbium and Columbium Alloy Bar, Rod, and Wire (B 392 – 62 T),
Columbium and Columbium Alloy Strip, Sheet, Foil, and Plate (B 393 – 62 T), and
Columbium and Columbium Alloy Seamless and Welded Tubes (B 394 – 62 T).

Revision and Reversion to Tentative of Standard Specifications for:

Slab Zinc (B 6 – 58), and
Rolled Zinc (B 69 – 39).

The new and revised specifications are available as separate reprints.

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Tentatives Continued Without ***Revision***

Tentative Specifications for:

Refined Secondary Lead (**B 325 - 58 T**),
Classification of Pig Tin (**B 339 - 59 T**),
Solder Metal (**B 32 - 60 T**),
Rosin Flux Cored Solder (**B 284 - 60 T**),
Seamless Nickel and Nickel Alloy Condenser and Heat Exchanger Tubes (**B 163 - 61 T**),
Nickel-Chromium-Iron Alloy Rod and Bar (**B 166 - 61 T**),
Nickel-Chromium-Iron Alloy Seamless Pipe and Tube (**B 167 - 61 T**),
Nickel-Chromium-Iron Alloy Plate, Sheet, and Strip (**B 168 - 61 T**),
Factory-Made Wrought Nickel and Nickel-Base Alloy Welding Fittings (**B 366 - 61 T**),
Remelted Lithium Metal in Ingot Form (**B 357 - 60 T**),
Titanium and Titanium Alloy Strip, Sheet, and Plate (**B 265 - 58 T**),
Titanium Sponge (**B 299 - 58 T**),
Seamless and Welded Unalloyed Titanium Pipe (**B 337 - 58 T**),
Seamless and Welded Unalloyed Titanium Tubing (**B 338 - 61 T**),
Titanium and Titanium Alloy Bars and Billets (**B 348 - 59 T**),
Seamless and Welded Unalloyed Titanium Welding Fittings (**B 363 - 61 T**),
Titanium and Titanium Alloy Castings (**B 367 - 61 T**),
Titanium and Titanium Alloy Forgings (**B 381 - 61 T**),
Zirconium Sponge and Other Forms of Virgin Metal for Nuclear Applications (**B 349 - 60 T**),
Zirconium and Zirconium Alloy Ingots for Nuclear Application (**B 350 - 60 T**),
Hot Rolled and Cold Finished Zirconium and Zirconium Alloy Bars, Rod, and Wire for Nuclear Application (**B 351 - 60 T**),
Zirconium and Zirconium Alloy Sheet, Strip, and Plate for Nuclear Applications (**B 352 - 60 T**), and
Zirconium and Zirconium Alloy Forgings and Extrusions for Nuclear Applications (**B 356 - 60 T**).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Refined Copper (H. Barkell, chairman).—The subcommittee ballot on the draft ISO recommendation DR 530, Specifications for Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars proposed by ISO/TC 26, resulted in approval which was reported to the chairman of the Supervisory Group to ASA on ISO/TC 26.

A Joint Task Group (with Committee B-1 on Wires for Electrical Conductors) is studying annealability tests for electrolytic tough pitch copper.

A Joint Task Group (with Committee B-5 on Copper and Copper Alloys, Cast and Wrought) on Resistivity of DLP Copper is studying the proposal for three grades.

A request has been received from Committee B-5 for including in Specification B 170, coverage for billets of "certified oxygen-free copper." Committees B-5 and F-1 on Materials for Electron Tubes and Semiconductor Devices have been approached to set up with Committee B-2 a joint task group to clarify requirements for this grade and to select an appropriate designation.

Subcommittee II on Refined Lead, Tin, Antimony, and Bismuth (A. Y. Bethune, chairman).—The wording of the scope clause in the Specification for Pig Lead (B 29) continues to be in dispute.

Subcommittee III on Tin Base and Lead Base Alloys (P. R. White, chairman).—A committee letter ballot to adopt the Specification for Solder Metal (B 32) as standard resulted in a negative vote, based on objection to the present maximum permissible arsenic content (0.02 per cent) in all grades as unnecessary in many applications.

Subcommittee IV on Refined Zinc and Wrought Zinc (R. G. Redelfs, chairman).—The Specification for Slab Zinc (B 6) will require further modification in the section on chemical analysis.

The Task Group on Cadmium is awaiting action by the appropriate E committees on sampling and analysis.

The Task Group on Cast and Wrought Zinc Anodes has failed to resolve differences. A report will be submitted at the June meeting.

A request has been received from Committee B-6 on Die-Cast Metals and Alloys for an additional grade of zinc of higher purity to meet the requirements of the new No. 7 die-cast alloy.

Subcommittee V on Precious Metals (C. D. Coxe, chairman).—The task group working on silver specifications has been asked to prepare requirements for three grades. Two drafts have been considered dealing with the first grade covering 999 purity.

Subcommittee VII on Refined Nickel and Cobalt, High Nickel Alloys and High Cobalt Alloys, Cast and Wrought (W. P. McFatridge, chairman).—A task group is being appointed to prepare supplementary requirements for several nickel alloy specifications to cover commercial nuclear applications.

Preparation of drafts for 30 per cent nickel—21 per cent chromium—balance iron alloy is in progress.

Specifications for alloys of the Hastelloy X and Hastelloy F types are being considered for use in boiler codes.

Subcommittee VIII on Less Common Metals and Alloys (R. I. Jaffee, chairman).—The Task Group on Molybdenum is engaged in incorporating new molybdenum alloys in the present group of molybdenum mill products specifications which were accepted last year.

The Task Group on Tungsten has submitted for subcommittee ballot a new Tentative Specification on Tungsten Billets, Bars, Rods, and Preforms for Forging and is revising the specification in accordance with the response received.

The Task Group on Uranium has completed four specifications for normal and enriched uranium melting stock and castings. These were balloted and are now being revised in accordance with the response received.

Subcommittee IX on Titanium and Titanium Alloys (A. F. Jones, chairman).—Task groups are working on revisions to include new alloys in tentative specifications under jurisdiction of the subcommittee.

Subcommittee X on Zirconium and Hafnium (W. W. Stephens, chairman) has prepared for committee ballot revisions of all six zirconium tentative specifications under the jurisdiction of the subcommittee.

A new tentative specification for hafnium sponge will be submitted to committee letter ballot.

This report has been submitted to letter ballot of the committee, which consists of 144 members; 138 members returned their ballots, of whom 127 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

ALFRED BORNEMANN,
Chairman.

C. K. CONARD,
Secretary.

REPORT OF COMMITTEE B-3
ON
CORROSION OF NONFERROUS METALS AND ALLOYS

Committee B-3 on Corrosion of Nonferrous Metals and Alloys held two meetings during the year: in New York, N. Y. on June 25, 1962, and in Montreal, Canada, on Feb. 4, 1963.

The committee consists of 84 members, 83 of whom are voting members; 29 are classified as producers, 29 as consumers, and 25 as general interest members.

Subcommittee chairmen have been designated as follows: Subcommittee I on Total Immersion and Subcommittee II on Alternate Immersion, D. H. Thompson to replace A. W. Tracy; Subcommittee III on Salt Spray, W. D. McMaster to replace C. O. Durbin; Subcommittee VI on Atmospheric Corrosion, T. J. Summerson to replace J. S. Pettibone; Subcommittee VII on Weather, P. J. Sereda to replace C. P. Larrabee; and Subcommittee VIII on Galvanic and Electrolytic Corrosion, J. J. Casey to replace G. V. Kingsley.

RECOMMENDATIONS AT ANNUAL MEETING

Tentatives Continued Without Revision

The committee recommends the continuation without revision of the following tentatives:

Tentative Methods of:

Total Immersion Corrosion Test of Nonferrous Metals (**B 185 – 43 T**),

Alternate Immersion Corrosion Test of Nonferrous Metals (**B 192 – 44 T**), and

Corrosion Testing of Decorative Chromium Plating by the Corrodkote Procedure (**B 380 – 61 T**).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee VI on Atmospheric Corrosion (T. J. Summerson, chairman).—A reprint, "Atmospheric Exposure of Nonferrous Metals and Alloys," has been issued which includes the 0-, 1-, and 2-yr data for the 77 alloys exposed in the 1957 test program. These data were reported in the Society's *Proceedings* for 1959, 1961, and 1962.[1]

The proposal for temporary removal for corrective processing of certain test panels which, on visual inspection, had shown lamellar edge splitting or exfoliation attack was rejected by the subcommittee since it was considered that removal and re-exposure introduced unknown test variables. The subcommittee did authorize the permanent removal from test of a number of test panels at the Point Reyes, Calif., site which apparently had been removed from test racks by vandals and left on the ground.

In the report of the 1957 test program the subcommittee authorized expanding

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 59, p. 176 (1959); Vol. 61, p. 205 (1961); and Vol. 62, p. 216 (1962).

Footnote *d* to Table II for the 2-yr test data to read: "Intergranular attack. (The indications of intergranular attack should not be construed in a general way that the items so marked would be unserviceable. Much longer exposures to these environments will be required to determine if the susceptibility to intergranular attack will develop exfoliation, and a stress corrosion test would be required to evaluate stress corrosion tendencies.)"

A visual inspection of the 7-yr specimens on exposure was made by a task group and reported to the subcommittee. This inspection is appended to this report.[2]

The deeper pitting of zinc panels after two years in the present test as compared with an earlier test is believed to be due to the fact that the present panels are exposed at 30 deg from the horizontal as compared with vertical exposure for the earlier program.

Subcommittee VII on Weather (P. J. Sereda, chairman).—The subcommittee has agreed to cooperate with the Frankford Arsenal in the calibration of marine, sunlight, and jungle sites in the Panama Canal Zone. Preliminary work will involve the installation of dew collectors and the measurement of the atmospheric chlorides, atmospheric SO_2, panel temperature, and time-of-wetness factors.

The Task Group on Measurement of Atmospheric Factors Affecting the Corrosion of Metals reported that specimens of steel, zinc, and copper were being exposed at each of six test sites each month for a one-month period—the program ending in March, 1963.

Analysis of the monthly data obtained suggests that correlations exist between the corrosion of panels and the atmospheric factors being studied (time of panel wetness, panel temperature during time of wetness, and atmospheric SO_2 activity during time of wetness).

The determination of atmospheric chlorides is underway at the two Kure Beach, N. C., test sites.

Subcommittee VIII on Galvanic and Electrolytic Corrosion (John J. Casey, chairman) issued the third progress report on the magnesium panel-type galvanic couples exposed at four locations as appended to this report.[3]

This report has been submitted to letter ballot of the committee, which consists of 83 voting members; 59 members returned their ballots, of whom 55 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

K. G. COMPTON,
Chairman.

W. H. AILOR,
Secretary.

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[2] See p. 168.

[3] See p. 170.

APPENDIX I

REPORT OF SUBCOMMITTEE VI ON ATMOSPHERIC CORROSION

1957 TEST PROGRAM

KURE BEACH 80-FT SITE, VISUAL INSPECTION, JUNE 6, 1963

The panels of the 1957 Test Program were inspected on June 6, 1963, the half way point between the 2- and 7-yr period removals. Inspection was made under the supervision of E. A. Tice, chairman of Inspection Committee. Three specimens of each alloy were inspected and their average conditions is reported below:

Alloy	Condition
No. 10	Aluminum colored, Slight etch.
No. 11	Aluminum colored, Medium etch.
No. 12	Aluminum colored, Light etch.
No. 13	Aluminum colored, Medium etch.
No. 14	Aluminum colored, Light etch.
No. 15	Aluminum colored, Light etch.
No. 16	Aluminum colored, Medium etch.
No. 17	Aluminum colored, Light etch.
No. 85	Aluminum colored, Medium etch.
No. 86	Aluminum colored, Medium etch.
No. 18	Aluminum colored, Medium etch.
No. 19	Aluminum colored, Severe etch.
No. 20	Aluminum colored, Medium etch.
No. 21	Aluminum colored, Medium etch.
No. 22	Aluminum colored, Medium etch.
No. 23	Aluminum colored, Medium etch.
No. 24	Aluminum colored, Light etch.
No. 25	Aluminum colored, Light etch.
No. 26	Aluminum colored, Light etch.
No. 27	Aluminum colored, Light etch.
No. 28	Gray colored, Heavy etch.
No. 29	Aluminum colored, Medium etch.
No. 84	Aluminum colored, Light etch, lined pattern.
No. 30	Aluminum colored, Medium etch.
No. 31	Severe exfoliation blistering on two of three panels examined, all over surface. 3rd panel exhibits exfoliation along one edge only.
No. 32	Aluminum colored, Severe etch.
No. 33	Aluminum colored, Severe etch.
No. 34	Aluminum colored, Medium etch.
No. 35	Aluminum colored, Light etch.
No. 36	Aluminum colored, Light etch.
No. 37	Aluminum colored, Medium etch.
No. 38	Aluminum colored, Medium etch.
No. 39	Aluminum colored, Medium etch.
No. 87	Aluminum colored, Light etch.
No. 40	Gray color with white spangled pattern. Surface smooth.
No. 41	Uniform khaki-colored stain over lead-colored surface. Surface smooth.
No. 42	Uniform white film, smooth. Multitudes of pits; more numerous near edges.
No. 43	Smooth slate gray color, numerous pits.
No. 44	Smooth uniform light cocoa-colored film, plus scattered and frequent pits.
No. 45	Rough granular surface, light gray colored.
No. 46	Severe pitting all over, dark gray color.
No. 47	Smooth, uniform "mink brown" color.
No. 48	Gray, metallic, smooth surface.
No. 49	Smooth surface, medium amount of rust stain, especially near edges.
No. 50	Smooth, metallic gray color.
No. 51	Uniform light green patina.
No. 52	Mottled effect, some rust stain from scattered pits.
No. 53	Smooth, very metallic color.
No. 54	Smooth, very metallic color.
No. 55	Slight brownish shade color, plus incipient pitting.
No. 56	Mostly metallic, with some brownish stain.
No. 57	Like No. 56, but lighter brown stain.
No. 58	Like No. 56, but slightly darker brown stain.
No. 59	Smooth, mostly metallic, with occasional pinpoint discoloration.
No. 60	Same as No. 59.
No. 61	Rough surface, uniform white corrosion product, panels warped.
No. 62	Rough surface, uniform white corrosion product over a blue-gray base.

Alloy	Condition
No. 64	Metallic sheen.
No. 65	"Mink brown" color, slight etch.
No. 66	Greenish brown color, medium etch.
No. 67	Maroon colored film over greenish film, heavy etch.
No. 68	Dark maroon, smooth surface.
No. 69	Tan film, smooth surface.
No. 70	Brown-maroon film, smooth surface.
No. 71	Brown-maroon film, smooth surface.
No. 72	Brown film in center, green near edges, smooth.
No. 73	Brown film in center, green near edges, smooth.
No. 74	Uniform brown film, smooth surface.
No. 75	Dark brown, mottled with greenish white, slight etch.
No. 76	Same as No. 75.
No. 77	Same as No. 75.
No. 78	Greenish brown, with green near edges. Slight etch.
No. 79	Greenish brown, slight green near edges. Slight etch.
No. 80	Purple, mottled with gold beige color, medium etch.
No. 81	Brown colored center, light green near edges, smooth.
No. 82	Brown colored center, light green near edges, smooth.
No. 83	Brown colored center, light green near edges, smooth.

Respectfully submitted on behalf of the subcommittee.

T. J. SUMMERSON,
Chairman.

APPENDIX II

REPORT OF SUBCOMMITTEE VIII ON GALVANIC AND ELECTROLYTIC CORROSION

Part III—Magnesium Panel-Type Galvanic Couples

The second set of magnesium panel-type galvanic couples was removed from the Kure Beach, N. C., 80-ft site on Dec. 14, 1960, after 2 years' exposure. The second set of similar panels exposed in the New York Area, State College, Pa., and the Kure Beach, N. C., 800-ft site will not be removed until after 5 years' exposure.

The initial report on this project[1] outlines the general program and procedures followed in preparing the panels. It also described the processing of the first set of panels removed after 1 year's exposure at four exposure sites. The same processing procedures were employed in the evaluation of the 2-year set of panels (56 couples) from the Kure Beach 80-ft site.

Table I summarizes the contact resistance between the magnesium panel and the dissimilar metal coupled to it. These measurements were made shortly after removal of the panels from test.

After disassembly of the couples, the magnesium alloy panels were cleaned in hot (200 F) 20 per cent chromic acid to remove all corrosion product. The extent of galvanic attack on each of these panels was then visually evaluated in terms of an arbitrary 1 to 5 scale where "1" represented the least, and "5" the greatest amount of galvanic attack. The results of this evaluation are presented in Table II which reveals the relative effect of the various dissimilar metals on the galvanic corrosion of AZ31B and ZK60A magnesium alloys. The dissimilar metals are listed according to increasing galvanic effect.

Tensile data obtained on specimens machined from the cleaned panels are summarized in Table III. The difference between the tensile strength of the control panels and the tensile strength of the panels to which the various dissimilar metals were coupled was intended to be a measure of the severity of the galvanic attack suffered by the magnesium panels.

In general, both the visual observations and the tensile data on the 2-year set of panels substantiated the relative galvanic effect of the various metals as determined from similar data on the 1-year set of panels exposed at the Kure Beach 80-ft site. Most galvanic corrosion occurred within the first year of exposure. The seven aluminum alloys caused the least galvanic corrosion when coupled to either AZ31B or ZK60A. The magnesium alloys experienced the severest galvanic attack when in contact with brass, monel, mild steel, and stainless steel. Chromium

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[1] Report of Subcommittee VIII on Galvanic and Electrolytic Corrosion, *Proceedings*, Am. Soc. Testing Mats., Vol. 61, p. 215 (1961).

TABLE I.—CONTACT RESISTANCE BETWEEN DISSIMILAR METAL AND BASE PANEL AFTER TWO YEARS' EXPOSURE AT THE 80-FT SITE, KURE BEACH, N. C.

Couple	Dissimilar Metal (Strip B)	Resistance, ohms[a]	
		AZ31 Panels[b]	ZK60 Panels[c]
No. 1	99.99 per cent Aluminum	Nil	Nil
No. 2	99.99 per cent Aluminum + 2 per cent Magnesium	Nil	Nil
No. 3	5356 Aluminum	Nil	Nil
No. 4	6061 Aluminum	Nil	Nil
No. 5	5052 Aluminum	Nil	Nil
No. 6	Alclad 7075 Aluminum	Nil	Nil
No. 7	Mild steel	5000	0.5, 2000
No. 8	85-15 brass	Nil	1.5
No. 9	Type 304 stainless steel	1.6	0.6, 1.4
No. 10	Chrome plated type 304 stainless steel	1.4	2000
No. 11	Monel	2000	1600, 5000
No. 12	5356 Aluminum	Nil	Nil
No. 13	Alcan B54S	Nil	Nil
No. 14	AZ31 or ZK60 (6061 Strip A)	Nil	Nil
No. 15	AZ31 or ZK60 (AZ31 or ZK60 Strip A)	>100 000[d]	>100 000[d]
No. 16	AZ31 or ZK60 (5356 Strip A)	Nil	Nil

[a] Represent the average contact resistance, except where noted, measured between strip B and base panel on both skyward and groundward surfaces.

[b] 1 panel per test.

[c] 2 panels per test.

[d] Creep of plastic bolts resulted in a loose assembly.

TABLE II.—EFFECT OF VARIOUS DISSIMILAR METALS ON THE GALVANIC CORROSION OF AZ31 AND ZK60 AFTER TWO YEARS' EXPOSURE AT THE 80-FT SITE, KURE BEACH, N. C.

Galvanic Attack Rating[a]	AZ31 Panels	ZK60 Panels
1	AZ31[b]	ZK60[b]
2	99.99 per cent Aluminum 99.99 per cent Aluminum + 2 per cent Magnesium 5356 Aluminum[b] 6061 Aluminum 5052 Aluminum Alcan B54S[d] Aluminum	99.99 per cent Aluminum 99.99 per cent Aluminum + 2 per cent Magnesium 5356 Aluminum[c] 6061 Aluminum 5052 Aluminum Alcan B54S[d] Aluminum
3	Alclad 7075 Aluminum	Alclad 7075 Aluminum Chrome plated type 304 stainless steel
4	Chrome plated type 304 stainless steel	
5	Type 304 stainless steel Monel Mild steel 85-15 brass	Type 304 stainless steel Monel Mild steel 85-15 brass

[a] Based upon visual examination after cleaning of panels:
1 = No significant galvanic corrosion
2 = slight galvanic attack, some shallow pits,
3 = moderate attack, scattered medium deep pitting,
4 = moderate attack, continuous band of medium deep pitting, and
5 = heavy attack, continuous band of deep pitting.

[b] Includes three different combinations of fastener materials:
(*a*) 6061 strip A + 6061 bolts.
(*b*) AZ31 (or ZK60 in ZK60 panels) strip A + plastic bolts.
(*c*) 5356 strip B + 5056 rivets.

[c] Includes two different combinations of fastener materials:
(*a*) 6061 strip A + 6061 bolts.
(*b*) 5356 strip A + 5056 rivets.

[d] Similar to AA5083 composition.

TABLE III.—TENSILE DATA ON AZ31B AND ZK60A PANELS AFTER TWO YEARS' EXPOSURE AT THE 80-FT SITE AT KURE BEACH, N. C.

Couple	Metal Coupled to AZ31B or ZK60A			AZ31B Panels			ZK60A Panels		
	Fastener[f]	Strip A[f]	Strip B[f]	Tensile Strength, psi[a]		Per Cent Loss in Tensile Strength[b]	Tensile Strength, psi[c]		Per Cent Loss in Tensile Strength[b]
				Avg	Range		Avg	Range	
No. 1	6061	6061	99.99 per cent Aluminum	39 960	170	0.5	48 860	560	1.3
No. 2	6061	6061	99.99 per cent Aluminum + 2 per cent Magnesium	38 960	850	3.0	48 850	660	1.3
No. 3	6061	6061	5356	39 430	330	1.8	48 920	740	1.2
No. 4	6061	6061	6061	38 650	140	3.8	48 550	2 290	1.9
No. 5	6061	6061	5052	39 000	230	2.9	49 420	690	0.2
No. 6	6061	6061	Alclad 7075	38 310	110	4.6	48 140	1 070	2.8
No. 7	Mild Steel	Mild Steel	Mild Steel	32 250	1350	19.7	28 260	24 520	42.8
No. 8	Brass	Brass	Brass	32 480	3340	19.1	20 820	21 510	58.0
No. 9	No. 304 Stainless Steel			34 910	1030	13.1	35 160	11 960	29.0
No. 10	Chrome Plated No. 304 Stainless Steel			38 030	3310	5.3	48 400	1 910	2.2
No. 11	Monel	Monel	Monel	34 020	440	15.3	31 810	21 480	35.7
No. 12	5056 Rivets	5356	5356	38 670	280	3.7	48 600	2 090	1.8
No. 13	5056 Rivets	5356	Alcan B54S	38 870	570	3.2	47 690	1 770	3.7
No. 14	6061	6061	AZ31B/ZK60A[d]	40 480	680	+0.8	49 840	650	+0.7
No. 15	Plastic	AZ31B/ZK60A[d]	AZ31B/ZK60A[d]	39 960	510	0.5	49 660	400	+0.3
No. 16	5056 Rivets	5356	AZ31B/ZK60A[d]	40 900	460	+1.8	49 440	600	0.1
No. 17	Control (No Couple)[e]			40 170	1230	...	49 500	1 300	...

[a] Based on average of 3 specimens from 1 panel for Couples 1 to 16 and 16 specimens from 4 panels for Couple 17.

[b] Based on difference between average tensile strength of uncoupled control panels (Couple 17) and panels coupled to various metals.

[c] Based on average of 6 specimens from 2 panels for Couples 1 to 16 and 16 specimens from 4 panels for Couple 17.

[d] AZ31B strip with AZ31B panel, and ZK60A strip with ZK60A panel.

[e] Initial tensile properties based on average of 12 bars from 4 unexposed panels per alloy.

Alloy	Thickness, in.	Elongation, per cent	Tensile Strength, psi
AZ31B-H24	0.188	19.5	40 500
ZK60A-T5	0.202	14.5	49 900

[f] Aluminum alloy designated except when otherwise noted.

plating on stainless steel was very effective in reducing galvanic corrosion caused by stainless steel alone. ZK60A suffered greater galvanic attack than AZ31B when coupled to brass, monel, mild steel, and stainless steel, but there was no significant difference in galvanic attack when these alloys were coupled to the other dissimilar metals.

Respectfully submitted on behalf of the subcommittee,

J. J. Casey,
Chairman.

REPORT OF COMMITTEE B-4

ON

METALLIC MATERIALS FOR THERMOSTATS AND FOR ELECTRICAL RESISTANCE, HEATING, AND CONTACTS*

Committee B-4 on Metallic Materials for Thermostats and for Electrical Resistance, Heating, and Contacts held three meetings during the year: in Pittsburgh, Pa., on Feb. 6, 1962; in New York, N. Y. on June 26, 1962; and in Washington, D. C. on Nov. 20, 1962.

The committee consists of 85 members, of whom 30 are classified as producers, 35 as consumers, and 20 as general interest members.

The committee is cooperating with the University of Maine at Orono, Maine, in continuing the seminars on electrical contacts which were previously held at The Pennsylvania State University in University Park, Pa. This year's symposium was held June 10 to 12, 1963.

Committee B-4 is cooperating with Peter Klaudy of the University of Graz, Austria, in arranging for an international symposium on electrical contacts to be held in May, 1964.

The committee continues to sponsor the *Bibliography and Abstracts on Electrical Contacts*, published as *ASTM STP 56-G* with annual supplements. In 1962, the Society published Supplement *ASTM STP 56-P* covering the literature and abstracts for 1961. The committee also sponsored the *Bibliography and Abstracts on Thermostat Metals, ASTM STP 288*, and the Supplement *ASTM STP 288-A* for 1960–1961.

Committee B-4 had special speakers at two of its meetings. At the June meeting, Dimitry Grabbe of Photocircuits, Inc., gave a talk on "Properties of Materials and Sliding Contact Problems"; and at the November meeting, W. J. Youden of the National Bureau of Standards gave a talk on "Statistical Analysis of Experimental Data and the Design of Experiments," and R. M. Sears of Texas Instruments, Inc., gave a talk on "Fundamentals of Thermostat Metals,"

There were several changes in the officers of the committee and the subcommittees during the past year. C. K. Strobel, secretary of Committee B-4, retired and was honored at a special luncheon at the February meeting in Pittsburgh. R. M. Sears was appointed by the Advisory Committee to be secretary and has had the help of Mr. Strobel in the transfer of his duties. C. W. Armstrong resigned as chairman of Subcommittee I although remaining as a member and was replaced by Jack Carlson. Merle Swinehart resigned as vice-chairman of Subcommittee IV although remaining as a committee member, and his activities have been taken over by John Sanders. Mr. Strobel was also secretary of Subcommittee IV and these duties have been assumed by Merle Elliott.

The subcommittees have reviewed all the standards and tentatives under their jurisdiction as required, and there are no recommendations at this time.

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Electrical Heating and Resistance Materials (Jack Carlson, chairman) was reorganized following the November, 1962, meeting. The subcommittee is considering increasing its activities by adding new members in various areas of application of resistance wire. Considerable work has been done on the uniformity of resistance wire and means of measuring this characteristic. Consideration is being given to specifications for sheathed heating wire applications.

Subcommittee III on Thermostat Metals (U. U. Savolainen, chairman) is continuing with the basic activities concerned with flexivity of thermostat metals, the thermal emissivity of metals either in vacuum or in air, thermal conductivity, corrosion, mechanical torque rate of coils, thermal deflection rate of coils, and the thermal and mechanical stability of thermostat metals.

The Bibliography and Abstracts on Thermostat Metals was published during this year as *ASTM STP 288*. The activities mentioned above are being considered for further improvement of present standards and the development of new standards.

Subcommittee IV on Contact Materials (J. D. Kleis, chairman):

Section A on Life Tests.—A new device change for contact welding studies with controlled bounce has been developed by N. C. Shaw. Several devices have been built, and tests run in different laboratories on this device indicate reasonable initial correlation. This program originally started with copper contacts and will continue with other metals of known composition and origin.

Section B on Physical Properties.—C. R. Marsland has prepared a critique of certain procedures used in physical testing of wire contact materials which will be helpful in the review of present specifications and the writing of future ones; when finished, it will be transmitted to other committees in the Society for their consideration.

Section C on Definition of Terms continues to define terms relative to contact operation and construction. This list has been coordinated with the Engineers Joint Council *Thesaurus*.

Section D on High Current Contacts is working to define the situation under which contacts should be tested under heavy currents so that the information will be suitable over a large range of heavy current application.

Section E on Bibliography.—The *ASTM Bibliography on Electrical Contacts* has been published and is available as *ASTM STP 56-P*.

Section F on Microcontacts is investigating the effect of high and low humidities upon the performance of the microcontact tester and the resistance of the contacts. It appears that there is some effect on the tester under high humidity conditions. Further testing will be done on 24 carat gold and gold-silver alloy contacts.

Section G on Static Connectors and Connections.—The crossed wire contacts which have been exposed since August, 1960, have been discontinued because of the deterioration of the insulation. A full report on the results of these tests will be made available shortly.

Section H on Sliding Contacts continues to work on standardizing a test procedure for measuring the characteristics of wire sliding contacts against metal slip rings. Standardization of the slip rings, wire, contact force, and speeds are being carried out. The purposes are to select the means of measurement for the specification of sliding contact combinations, to develop test methods for measuring special properties of sliding contact materials, and to develop a test procedure for measuring wear and noise.

This report has been submitted to letter ballot of the committee, which consists of 85 members; 45 members returned their ballots, of whom 42 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

ERLE I. SHOBERT, II,
Chairman.

R. M. SEARS,
Secretary.

REPORT OF COMMITTEE B-5

ON

COPPER AND COPPER ALLOYS, CAST AND WROUGHT*

Committee B-5 on Copper and Copper Alloys, Cast and Wrought, held a meeting at Niagara Falls, Ontario, Canada, in September, 1962, and is scheduled to meet at Washington, D. C., in April, 1963. At the September meeting, Subcommittees W-1, W-2, W-3, W-4, F-1, G-1, and G-3, and several task groups met; and there was a group dinner and tour of Sir Adam Beck Generating Station. Also, Subcommittee W-4 and the U.S.A. National Committee for ISO/TC-26 and task groups met in June, 1962, during the Annual Meeting. At the April, 1963, meeting it is planned that the above mentioned subcommittees and ISO group will meet, and that a paper on "The Influence of Composition, Strain Hardening, and Precipitation Heat Treatment on the Mechanical Properties and Stress Corrosion Resistance of Copper-Beryllium Strip," by Messrs. G. R. Gohn, G. J. Herbert, and J. B. Kuhn, will be presented by Mr. Gohn.

The committee consists of 106 voting members; 43 are classified as producers, 35 as consumers, and 28 as general interest members.

At the 1962 Annual Meeting, an ASTM Award of Merit was conferred on W. D. France. K. B. Bissell was appointed as B-5 representative to Committee E-8 on Nomenclature and Definitions. Also, Mr. Bissell served on a special task group to formulate a proposal on Decimal System for Numbering Paragraphs in ASTM Specifications.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1962 Annual Meeting, Committee B-5 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specification for:

U-Bend Seamless Copper and Copper Alloy Heat Exchanger and Condenser Tubes (B 395 - 62 T).

Revision and Reversion to Tentative of Standard Specifications for:

Seamless Brass Tube (B 135 - 61), and

Seamless Copper and Copper-Alloy Rectangular Waveguide Tube (B 372 - 61).

The recommendations were accepted by the Standards Committee on October 17, 1962, and the new and revised specifications appear in the Compilation of ASTM Standards on Copper and Copper Alloys, December, 1962.

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

RECOMMENDATIONS AFFECTING STANDARDS

The committee is recommending one new standard and revision of 5 standards for immediate adoption, and the adoption of 3 tentatives as standards. The standards affected, together with the revisions recommended, are covered in detail in the Appendix to this report.[1]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Executive Subcommittee (W. H. Jennings, chairman).—A joint Committee B-5 task group was set up to study CABRA Standard Designations for Coppers and Copper Alloys for possible use in B-5 specifications. Study of proposals for a new standard on sampling of copper and copper alloys for chemical analysis, and for consolidation of Methods E 75 and E 76 was assigned to the joint task group which is considering CABRA proposals on impurities. Joint task groups also are studying proposals on revision of electrical resistivity requirements for DLP tube, and surface oxidation of oxygen-free copper. There has been no further communication from the Industrial Fasteners Inst. so it was decided to drop consideration of a specification on fasteners. Also, it was decided that there was not enough interest to justify Committee B-5 liaison with Committee E-1 Task Group on Linear Expansion Test, and with the Special Committee on Numerical Data. Use of a unified numbering system for business items throughout Committee B-5 work is well under way and is proving very helpful.

Subcommittee W-1 on Plate, Sheet, and Strip (A. E. Moredock, chairman) recommended for immediate adoption a new Standard Specification on Copper-Nickel Alloy Plate and Sheet for Pressure Vessels (V. P. Weaver, chairman of task group).

Work under way includes: (1) the development of new specifications for copper foil, sheet, and strip for printed circuits; strip for flexible hose; thin strip with limits on maximum grain size; copper-nickel-silicon sheet and strip; and copper-alloy clad steel plate, with liaison from Committee A-1, Subcommittee XI, and (2) revision of Specifications B 36, B 122, and B 131 to add limits on impurities; B 100 as regards mechanical requirements; B 122 to add 90-10 copper-nickel and requirements for annealed copper-nickel alloys and change iron limits for 80-20 copper-nickel; and B 248 as regards sampling and retests.

Subcommittee W-2 on Rods, Bars, and Shapes (O. A. Klema, chairman) recommended for immediate adoption revision of Specification B 151, to add leaded 12 per cent copper-nickel-zinc, as alloy F (P. B. Tursi, chairman of task group).

Work under way includes: (1) the development of new specifications for low-leaded brass rod, and copper-nickel-silicon rod and bar; and (2) the revision of Specifications B 16, Tables I and II; B 151 to include hardness requirements, and to add 60-40 and 70-30 copper-nickel alloys; B 124 and B 283 to include tellurium-copper; and B 196 for general improvement.

Subcommittee W-3 on Wire and Wire Rod (R. H. Schoemann, chairman).—Work under way includes: (1) the development of a new specification for copper-nickel-silicon alloy wire; and (2) the revision of Specifications B 134 to add limits on impurities; B 159 to include square wire; and B 197 for general improvement.

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[1] See p. 181.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee W-4 on Pipe and Tube (J. M. Keyes, chairman) prepared (1) the new Tentative Specification for U-Bend Seamless Copper and Copper Alloy Heat Exchanger and Condenser Tubes (B 395 - 62 T) (W. D. France, chairman of task group), and (2) revisions of Specifications B 135 to add 63-37, and 65-35 brass alloys (V. P. Weaver chairman); and B 372 to add requirement to control surface finish because of its effect on micro attenuation (R. H. Schoemann, chairman). Also, the latest revisions of Specifications B 42, B 43, B 68, B 75, B 88, B 135, B 251, B 280, B 302, B 306, B 315, B 359, B 360, B 372 were recommended for possible adoption by ASA as American Standards; and Tentative Specifications B 135 and B 372 were recommended for adoption as standard.

Work under way includes: (1) the development of a specification for welded copper and copper alloy tube, and (2) revision of Specifications B 43, B 111, and B 135 to add limits on impurities; B 111, B 188, B 251, B 302, B 306, and B 315 as regards squareness of cut; B 315 to add alloy B of B 97; and B 359 to clarify several sections.

Subcommittee F-1 on Castings and Ingots for Remelting (M. L. Steinbuch, chairman) recommended revisions in Specifications B 61 and B 62 to include emission methods of chemical analysis; B 119 to revise wording defining "Brass vs. Bronze"; B 145 to change tin limits for Alloy 5B to 2.0 per cent min, 3.0 per cent max, to agree with change previously made in B 30; and B 147 to add a new section on pressure and fracture tests.

Work under way includes: (1) consideration of a specification for worm gear alloy, and (2) study of location of tension test specimens in castings.

Subcommittee G-1, on Methods of Test (P. D. Strubhar, chairman) recommended (1) a proposed revision of Method E 112 which was forwarded to Committee E-4, and (2) a proposed revision of Method E 8 which was forwarded to Committee E-1.

Work under way includes (1) development of methods of test for porosity in heavy gage shapes of oxygen-free copper, and for eddy current testing of tube; (2) consideration of continued reference to Method E 2 in Committee B-5 specifications, and (3) a procedure for measuring thickness.

Subcommittee G-2 on Tolerances (J. E. McGraw, chairman) continued its liaison work with CABRA.

Subcommittee G-3 on Editorial and Publications (K. B. Bissell, chairman).—Work under way includes further study of (1) classification of properties as to "Physical" versus "Mechanical" versus "Other"; (2) decimal system for numbering paragraphs in ASTM Specifications, (3) terms used in Committee B-5 specifications for inclusion in "Word List for Glossary of ASTM Definitions," and (4) consistency of Specifications B 248, B 249, B 250, and B 251 in sections on sampling and retests.

U.S.A. National Committee on ISO/TC-26 (W. H. Jennings, chairman) took action to advise ASA regarding letter ballot on seven final ISO Draft Recommendations covering methods of test for copper alloy products.

The work of U.S.A. National Committee for ISO/TC-26 was supported by the decision of the Board of Directors of CABRA to authorize $1000 annually for delegates to TC-26 meetings concerned with materials in CABRA's field.

V. P. Weaver assumed the assignment of Leader of ISO/TC-26 Working Group 4, for which U.S.A. holds the Secretariat. The group has developed Draft Recommendation No. 530, "Specifications for Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars," and is preparing three specifications covering fire refined and phosphorus deoxidized copper in refinery shapes.

Other work under way in ISO/TC-26 includes seven methods of test by Working Group 1; a classification of wrought copper (composition, manufactured forms, densities), mechanical properties of wrought copper, a classification of wrought copper alloys, and a uniform designation code for copper and copper alloy products, by Working Group 2; and a classification of cast copper and copper alloys, and preparation of test pieces for the determination of the mechanical properties of castings, by Working Group 3. Working Group 2 held a meeting at Thun, Germany, May 28-30, 1962. No U.S.A. delegates attended.

This report has been submitted to letter ballot of the committee, which consists of 103 voting members; 84 members returned their ballots, of whom 77 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. H. JENNINGS,
Chairman.

R. P. DESTITO,
Secretary.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS FOR COPPER AND COPPER ALLOYS, CAST AND WROUGHT

In this Appendix recommendations are given affecting certain specifications, both standard and tentative, referred to earlier in this report. The specifications appear in their present form in the 1961 Book of ASTM Standards, Part 2, or in the Compilation of ASTM Standards on Copper and Copper Alloys, December, 1962.

New Standard, Immediate Adoption

(requires nine-tenths affirmative vote)

Standard Specification for Copper Nickel Alloy for Pressure Vessels, as appended hereto.[3]

Adoption of Tentatives as Standards Without Revision

Tentative Specifications for:

Seamless Brass Tube (B 135 – 62 T),

Seamless Copper and Copper-Alloy Rectangular Waveguide Tube (B 372 – 62 T), and

U-Bend Seamless Copper and Copper Alloy Heat Exchanger and Condenser Tubes (B 395 – 62 T).

Revision of Standards, Immediate Adoption

(requires nine-tenths affirmative vote)

Standard Specification for Steam or Valve Bronze Castings (B 61 – 60):

Section 8.—Change the last sentence of Paragraph (*a*) to read: "For the purpose of analysis only, the chemical test coupon may consist of a tension test coupon or a separate chemical analysis test sample may be desired."

Add a new Paragraph (*c*) to read: "The chemical composition may also be determined by the emission methods, but should a dispute arise concerning the chemical composition, the wet chemical methods contained in ASTM Methods for Chemical Analysis of Metals shall be used as the umpire methods."

Reletter present Paragraphs (*c*) and (*d*) as Paragraphs (*d*) and (*e*).

Standard Specification for Composition Bronze or Ounce Metal Castings (B 62 – 60):

Section 8.—Change as indicated above for Section 8 of Specification B 61.

Standard Specification for Leaded Brass and Leaded Semi-Red Brass Sand Castings (B 145 – 61):

Table I.—Change tin value for alloy 5B from "3.00" to "2.50" per cent.

Table II.—Change tin value for alloy 5B from "2.5" minimum to "2.0" and change "3.5" maximum to "3.0."

Standard Specification for High-Strength Yellow Brass (Manganese Bronze) and Leaded High-Strength Yellow Brass (Leaded Manganese Bronze) Sand Castings (B 147 – 52):

Section 12.—Add a new Section 12 entitled "Pressure and Fracture Tests," to read as follows: "The castings shall conform to such requirements as to pressure and fracture tests as may be agreed upon by the manufacturer and the purchaser and as specified in the order."

Renumber present Sections 12 to 16 as Sections 13 to 17.

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[3] The new standard appears in the 1963 Supplement to Book of ASTM Standards, Part 2.

Standard Specification for Copper-Nickel-Zinc (Nickel Silver) Rod and Bar (B 151 – 58):

Section 1.—In Paragraph (*a*), change "six alloys" to read "seven alloys," and add letter "F."

Add Alloy "F" to the table with nominal composition of 62 per cent copper, 12 per cent nickel, 25 per cent zinc, and 1 per cent lead.

Change the last sub-paragraph of (*b*) to read "Alloys C and F."

Table I.—Add Alloy F having the following chemical requirements:

Copper	59.0 to 66.5
Nickel	11.0 to 13.0
Lead	0.8 to 1.4
Iron	0.50
Zinc	remainder

Table II.—Change first column subheading under "Tensile Strength" to read "Alloys A, C, and F."

Editorial Changes

Standard Specification for Brass Plate, Sheet, Strip and Rolled Bar (B 36 – 61):

Section 10(e).—Change to read as follows:

(e) Straightness:

(1) *Slit Metal.*—See Section 8(*e*), Table XI.

(2) *Slit Metal and Either Straightened or Edge-Rolled.*—See Section 8(*e*), Table XI.

(3) *Square-Sheared Metal.*—See Section 8(*e*), Table XII.

(4) *Sawed Metal.*—See Section 8(*e*), Table XIII.

Standard Specification for Copper-Silicon Alloy Plate, Sheet, Strip and Rolled Bar for General Purposes (B 97 – 55):

Section 7(e).—Change as indicated above for Section 10(*e*) of Specification B 36.

Standard Specification for Phosphor Bronze Plate, Sheet, Strip, and Rolled Bar (B 103 – 62):

Section 9(e).—Change as indicated above for Section 10(*e*) of Specification B 36.

Standard Specification for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 – 62):

Section 8.—In the Table of Section 8, transpose the last line "70-30 copper-nickel . . . 20" to appear opposite the temper designation "Drawn, stress relieved."

Table I and Section 16(e).—Delete the words "Type A" used to identify the 80-20 copper-nickel alloy.

Table II.—Delete the word "incl" in the column headed "Materials" for the last line under 70-30 copper-nickel.

In the column headed "Temper" repeat the line "drawn, stress-relieved" for both the 60-40 and 70-30 copper-nickel to indicate that the elongation values in the right hand column apply to the drawn, stress-relieved temper for both of these alloys.

Standard Specification for Leaded Brass Plate, Sheet, Strip, and Rolled Bar (B 121 – 60):

Section 10(e).—Change as indicated above for Section 10(*e*) of Specification B 36.

Standard Specification for Copper-Nickel-Zinc (Nickel Silver) and Copper-Nickel Alloy Plate, Sheet, Strip, and Rolled Bar (B 122 – 60):

Section 10(e).—Change as indicated above for Section 10(*e*) of Specification B 36.

Standard Specification for Copper Sheet, Strip, Plate, and Rolled Bar (B 152 – 60):

Section 14(e).—Change as indicated

above for Section 10(*e*) of Specification B 36.

Standard Specification for Aluminum Bronze Plate, Sheet, Strip, and Rolled Bar (B 169 – 55):

Section 7(e).—Change as indicated above for Section 10(*e*) of Specification B 36.

Standard Specification for Copper-Beryllium Alloy Plate, Sheet, Strip, and Rolled Bar (B 194 – 62):

Section 11(e).—Change as indicated above for Section 10(*e*) of Specification B 36.

Standard Specification for General Requirements for Wrought Copper and Copper-Alloy Plate, Sheet, Strip, and Rolled Bar (B 248 – 62):

Section 8(e).—Change the paragraph on Table XI to read as follows:

> *Table XI.*—Straightness tolerances for slit metal applicable to ASTM Designations B 36, B 97, B 103, B 121, B 122, B 152, B 169, and B 194. Straightness tolerances for slit metal and either straightened or edge rolled applicable to ASTM Designations B 36, B 97, B 103, B 121, B 122, B 152, B 169, and B 194.

Table XII.—Delete Table XII and renumber Tables XIII to XV as Tables XII to XIV.

Standard Specification for General Requirements for Wrought Copper and Copper-Alloy Rod, Bar, and Shapes (B 249 – 62):

Appendix.—In the Table of Standard Weights, add Alloy "F" with density of 0.318 lb per cu in. for ASTM Designation B 151.

REPORT OF COMMITTEE B-6
ON
DIE-CAST METALS AND ALLOYS*

Committee B-6 on Die-Cast Metals and Alloys held two meetings during the year: in New York, N. Y., on June 26, 1962, and in Washington, D. C., on Dec. 4, 1962.

The committee consists of 79 members, of whom 36 are classified as producers, 17 as consumers, and 26 as general interest members.

The committee donated $1500 to the Society's Building Fund from the earnings of the committee's Exposure Fund.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee B-6 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] The existing tentatives referred to in this report appear in their present form in the 1961 Book of ASTM Standards, Part 2.

Adoption of Tentatives as Standards With Revision

Tentative Specifications for Zinc-Base Alloy Die Castings (B 86 – 57 T):

Table I.—Change the chemical requirements for four of the elements in Alloy AG40A to read as follows:

Magnesium, per cent	0.020 to 0.05
Lead, max, per cent	0.005
Cadmium, max, per cent	0.004
Tin, max, per cent	0.003

Tentative Specifications for Zinc-Base Alloys in Ingot Form for Die Castings (B 240 – 57 T):

Table I.—Change the chemical requirements for four of the elements in Alloy AG40A to read as follows:

Magnesium, per cent	0.025 to 0.05
Lead, max, per cent	0.004
Cadmium, max, per cent	0.003
Tin, max, per cent	0.002

The committee also recommends that both revised specifications be submitted to the American Standards Association for adoption as American Standards.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Aluminum-Base Die-Casting Alloys (E. V. Blackmun, chairman) is planning to determine the effect of zinc on the elevated temperature properties of aluminum alloys SC84A and B. It is expected that data will be determined for two zinc contents, in the range of 1 to 3 per cent, and at three temperatures.

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

A report on the effect of iron contents on the mechanical properties of aluminum die-casting alloys has been prepared and is appended to this report.[2]

Also under study in the subcommittee is the advisability of raising the copper limit from 0.6 to 1.0 in aluminum alloys S12A and B.

Subcommittee II on Zinc-Base Die-Casting Alloys (A. E. Weiss, chairman) prepared the revisions in Specifications B 86 and B 240 noted earlier in this report and recommended that both revised specifications be adopted as standards and be submitted to the American Standards Association for adoption as American Standards.

The committee is still considering adding a new high-purity zinc die-casting alloy to Specifications B 86 and, subsequently, to B 240. Adoption of this alloy is being delayed because slab zinc of the required purity cannot be purchased from the grades specified in the Specifications for Slab Zinc (B 6). Committee B-6 has requested Committee B-2 on Nonferrous Metals and Alloys to consider means by which slab zinc of the purity required for the new alloy can be incorporated in their slab zinc specifications.

Opinions are being solicited regarding the advisability of retaining alloy AC41A in Specification B 86 and, if retained, whether the magnesium content and the impurity limits should be changed to conform to those of alloy AG40A.

Subcommittee V on Exposure and Corrosion Tests (D. H. Kleppinger, chairman) is studying the advisability of preparing and exposing anodically-coated die-cast test bars. The subcommittee is also compiling the published subnormal and elevated temperature properties for the zinc and aluminum die-casting alloys.

Subcommittee VII on Magnesium-Base Die-Casting Alloys (V. D. Sweeney, chairman) is considering including additional die-casting alloys in Specification B 94. Two alloys are currently being considered. These alloys are AM20XA containing 1.75 per cent aluminum, 0.3 per cent manganese, remainder magnesium characterized with good electrical and thermal conductivity properties; and K1A containing 0.7 per cent zirconium, remainder magnesium which is recommended for its damping properties.

This report has been submitted to letter ballot of the committee, which consists of 79 members; 74 members returned their ballots, of whom 71 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. BABINGTON,
Chairman.

GEORGE L. WERLEY,
Secretary.

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[2] See p. 186.

APPENDIX

EFFECT OF IRON CONTENT ON THE MECHANICAL PROPERTIES OF ALUMINUM DIE-CASTING ALLOYS

Report of Task Group of Subcommittee I on Aluminum and Aluminum Alloy Ingots

Tests were conducted to determine the effect of iron content in the range of 0.8 to 2.0 per cent on the mechanical properties of six commercial die-casting alloys. These tests showed that the ductility of the alloys as measured by per cent elongation was lowered as the iron content was raised. The decrease ranges from approximately 22 to 48 per cent. The yield strength of all alloys was raised, the maximum increase being approximately 13 per cent. The ultimate tensile strength was variably affected, with changes ranging from a loss of 5 per cent to a gain of 4 per cent.

Mechanical Property Tables

At the meeting of Subcommittee I on Aluminum-Base Die-Casting Alloys of ASTM Committee B-6 on Die-Cast Metals and Alloys, held in Cleveland, Ohio, on March 5, 1952, a suggestion was made to consider the inclusion of a tabulation of casting, finishing, and performance characteristics as an appendix to the Specification for Aluminum-Base Alloy Die Castings (B 85 – 50 T).[1] This proposal was then enlarged to include the suggestion that mechanical property tables already given be reviewed and revised, if necessary, to correlate with more recent data.

A task group consisting of Messrs. A. E. Martin, J. C. Fox, A. J. Prickett, and E. V. Blackmun (ex officio) was appointed and charged with preparing a table of alloy characteristics. This task group presented a report at the June 1952 meeting in New York City.

The table of mechanical properties submitted by the task group contained data for tensile strength, and 0.2 per cent offset yield strength, and per cent elongation, shearing strength, fatigue strength and Charpy impact strength for alloys G8A, S5C, S12A, S12B, SC54A, SC54B, SC84A, SC84B, SG-100A, and SG100B.

In the voting for the adoption of the table of mechanical properties, a number of negative ballots were cast, largely on the basis that:

1. The table contained data from only one producer, rather than an average of data from a number of producers.

2. The table contained values that were at variance with other accredited data, particularly with reference to the effect of iron content upon the properties.

At the March 3, 1953, meeting of the Subcommittee, the report was referred back for further study. At that time, Messrs. W. W. Broughton, D. L. Colwell, and D. H. Kleppinger were added to the task group to serve with the original members.

The task group meeting at the Apex Smelting Co. on Oct. 21, 1953, adopted a test plan to determine the properties of the alloys.

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[1] 1950 Supplement to Book of ASTM Standards, Part 2, p. 193.

Work Plan:

The concensus of the task group was that the only true solution to the problem of compiling a table of mechanical properties was to set up a cooperative test program in which a number of producers' castings would be evaluated. Representatives of the Doehler Jarvis Corp., The Precision Casting Co., and the Aluminum Company of America on the task group (Messrs. Fox, Martin, and Blackmun) offered the services of their firms for this purpose.

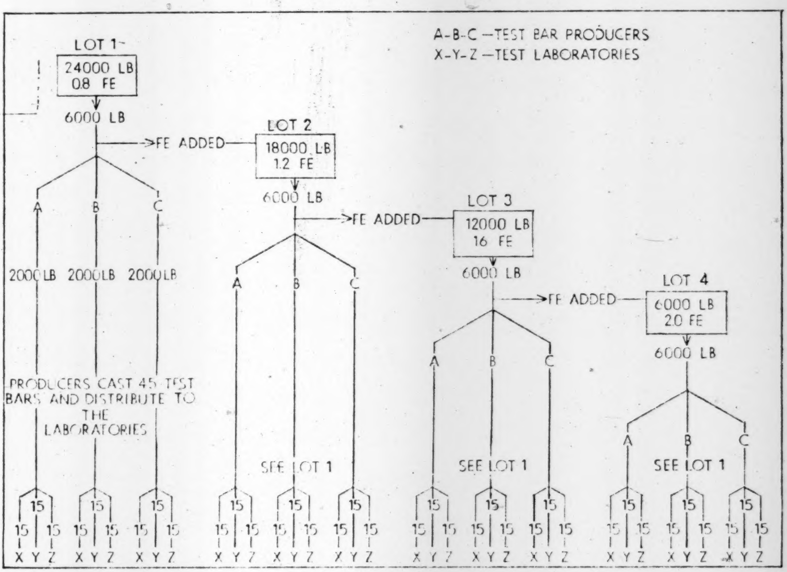
A-B-C —TEST BAR PRODUCERS
X-Y-Z —TEST LABORATORIES
LOT 1
24000 LB
0.8 FE
6000 LB
FE ADDED
LOT 2
18000 LB
1.2 FE
6000 LB
FE ADDED
LOT 3
12000 LB
1.6 FE
6000 LB
FE ADDED
LOT 4
6000 LB
2.0 FE
6000 LB
A
B
C
2000 LB
2000 LB
2000 LB
PRODUCERS CAST 45 TEST BARS AND DISTRIBUTE TO THE LABORATORIES
SEE LOT 1
SEE LOT 1
SEE LOT 1
15
X Y Z

FIG. 1.—Flow Sheet for Preparation, Distribution, and Testing of Die-Cast Aluminum Test Bars with Various Iron Contents.

It will be noted that Specification B 85 – 52 T[2] contained a total of ten alloy compositions, but that four of these, SC54, SC84, S12, and SG100, were present in modifications lettered "A" and "B," their difference being only in the maximum iron impurity limits. The iron limit of modification A was 1.3 per cent, whereas the iron limit for variety B was 2.0 per cent. If the A and B varieties are considered the same alloy with a large iron-content spread, only six alloys are really present.

Since questions had been raised over what constituted the iron content in a "typical" casting of both the hot-chamber and cold-chamber variety and of what effect iron content had on mechanical properties, the task group decided upon a test plan in which each of the six alloys would be investigated over an iron-content range of 0.8 per cent to 2.0 per cent.

During the course of the investigation, alloy SC54 was removed from Specification B 85, because of obsolescence. Alloy SC114 was added to the specification and was studied in this program.

The test plan for the program can be readily visualized by referring to Fig. 1. For each of the alloys studied, SC84, SC114, S5, S12, G8, and SG100, the Apex Smelting Co. prepared a master

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[2] Specification for Aluminum-Base Alloy Die Castings, 1952 Book of ASTM Standards, Part 2, p. 757.

24,000-lb heat of metal with average commercial impurities and a controlled iron content of 0.8 per cent. Of this heat, 6000 lb were tapped and the iron content in the balance was adjusted to 1.2 per cent, after which 6000 lb were again tapped and the iron content was adjusted to 1.6 per cent, and so forth. In this manner a 6000-lb heat of each alloy in each of four iron contents (0.8, 1.2, 1.6, and 2.0 per cent) was produced. These heats were divided into three lots of 2000 lb each and shipped to the three cooperating die casters. Alloy SC114 was produced in only three iron contents—0.8, 1.2, and 1.6 per cent. The chemical compositions of the heats produced are shown in Table I.

TABLE I.—CHEMICAL COMPOSITION OF SPECIAL HEATS OF ALUMINUM DIE-CASTING ALLOYS WITH CONTROLLED IRON CONTENTS.

Alloy Number Apex Heat	Chemical Composition, per cent[a]						
	Iron	Silicon	Copper	Magnesium	Manganese	Zinc	Nickel
S5C:							
No. 2380	0.80	4.99	0.47	0.03	0.23	0.33	...
No. 2381	1.16	4.98	0.58	0.03	0.23	0.33	...
No. 2382	1.52	5.19	0.59	0.03	0.24	0.34	...
No. 2383	1.90	5.08	0.57	0.03	0.23	0.34	...
S12A:							
No. 5263	0.82	11.64	0.56	0.07	0.28	0.27	0.04
No. 5264	1.23	12.00	0.57	0.05	0.29	0.28	0.05
No. 5265	1.62	12.05	0.58	0.04	0.29	0.29	0.05
No. 5266	1.89	11.98	0.57	0.04	0.30	0.30	0.05
SC84A:							
No. 5714	0.79	9.10	3.58	0.05	0.33	0.87	0.14
No. 5715	1.25	8.95	3.60	0.04	0.34	0.85	0.13
No. 5716	1.61	9.01	3.59	0.04	0.34	0.88	0.14
No. 5717	1.98	8.83	3.55	0.04	0.36	0.86	0.14
SC114A:							
No. 3350	0.80	11.23	3.49	0.05	0.39	0.87	0.36
No. 3357	1.18	10.96	3.43	0.03	0.39	0.81	0.34
No. 3358	1.57	10.83	3.41	0.03	0.39	0.83	0.34
SG100A:							
No. 2068	0.83	9.55	0.51	0.53	0.29	0.44	0.05
No. 2069	1.22	9.56	0.51	0.53	0.29	0.45	0.05
No. 2070	1.65	9.44	0.50	0.53	0.26	0.41	0.05
No. 2071	2.02	9.59	0.49	0.49	0.26	0.41	0.05
G8A:							
No. 2490	0.82	0.26	0.16	7.71	0.20	0.09	...
No. 4569	1.28	0.33	0.25	8.32	0.24	0.12	...
No. 4567	1.57	0.39	0.24	8.28	0.23	0.13	...
No. 4571	1.90	0.28	0.19	7.80	0.24	0.09	...

[a] Aluminum provides the balance of composition for each alloy.

Each die-casting firm produced a quantity of test bars, using the casting conditions believed to be typical for that alloy. Of those produced, 45 bars were distributed for each lot of metal, 15 each to the three testing laboratories: Bell Telephone Laboratories, The American Smelting and Refining

TABLE II.—MECHANICAL PROPERTIES OF DIE-CAST TEST BARS OF COMPOSITIONS WITH CONTROLLED IRON CONTENT.

Iron, per cent	Producer	Tensile Strength, psi				Yield Strength, psi				Elongation, per cent			
Laboratory.....		X	Y	Z	Average	X	Y	Z	Average	X	Y	Z	Average
Alloy S5C													
Lot 1:	A..	31 800	30 800	30 800	31 100	11 800	12 500	11 600	12 000	9.9	9.6	8.7	9.4
0.8........	B..	34 000	33 500	33 700	33 700	13 600	14 400	13 600	13 900	12.3	11.2	11.5	11.7
	C..	33 500	32 900	33 300	33 200	15 600	16 500	15 700	15 900	11.1	10.9	9.6	10.5
Average.....					32 700				13 900				10.5
Lot 2:	A..	31 700	30 400	30 800	31 000	12 000	12 500	11 600	12 000	8.9	7.9	7.2	8.0
1.2........	B..	34 500	34 200	34 000	34 200	14 800	14 700	14 700	14 700	10.6	10.9	10.6	10.7
	C..	34 100	33 100	33 600	33 600	17 100	16 200	17 800	17 000	9.7	9.5	8.7	9.3
Average.....					32 900				14 600				9.3
Lot 3:	A..	32 800	31 000	31 200	31 700	13 000	11 900	12 100	12 300	8.8	7.5	7.6	8.0
1.6.......	B..	34 600	34 600	34 600	34 600	14 500	15 100	14 600	14 700	10.4	10.5	10.3	10.4
	C..	32 600	32 600	33 300	32 800	15 600	15 000	15 700	15 400	7.9	9.0	8.6	8.5
Average.....					33 000				14 100				9.0
Lot 4:	A..	32 500	31 700	31 800	32 000	13 300	12 800	12 700	12 900	7.5	7.1	7.5	7.4
2.0.......	B..	35 200	35 400	35 100	35 200	14 800	14 900	14 900	14 900	9.1	9.9	9.6	9.5
	C..	33 400	33 400	34 300	33 700	16 900	16 500	16 700	16 700	7.0	8.1	7.9	7.7
Average.....					33 600				14 800				8.2
Grand Averages...					33 100				14 400				9.3
Alloy S12A													
Lot 1:	A..	43 500	42 300	41 600	42 500	18 800	18 800	18 100	18 600	4.1	4.7	4.4	4.4
0.8........	B..	43 600	43 000	43 400	43 300	19 200	19 400	19 400	19 300	5.0	5.1	4.4	4.8
	C..	41 700	40 200	42 200	41 400	19 900	20 500	20 900	20 400	3.7	3.6	3.6	3.6
Average.....					42 400				19 400				4.2
Lot 2:	A..	43 800	41 500	41 800	42 400	19 300	19 100	18 700	19 000	4.0	4.2	3.7	4.0
1.2........	B..	46 300	46 000	44 500	45 600	20 700	20 800	20 300	20 600	4.9	5.2	4.0	4.7
	C..	36 800	37 300	39 100	37 700	20 700	22 500	22 000	21 700	2.5	2.2	2.0	2.2
Average.....					41 900				20 400				3.6
Lot 3:	A..	43 000	41 500	42 700	42 400	19 800	19 700	20 500	20 000	3.0	3.1	3.5	3.2
1.6........	B..	46 800	47 600	47 500	47 300	22 700	23 800	22 500	23 000	3.8	4.8	3.8	4.1
	C..	42 200	43 400	39 500	41 700	21 700	23 100	21 600	22 100	2.8	3.1	1.8	2.6
Average.....					43 800				21 700				3.3
Lot 4:	A..	45 400	43 000	43 200	43 900	20 700	21 500	20 500	20 900	2.7	2.9	2.6	2.7
2.0........	B..	46 700	47 000	46 300	46 700	22 000	23 300	21 900	22 400	3.3	3.5	2.6	3.1
	C..	42 000	39 800	42 400	41 400	22 800	24 200	21 600	22 900	2.3	1.9	1.9	2.0
Average.....					44 000				22 000				2.6
Grand Averages...					43 000				20 900				3.4

TABLE II.—*Continued.*

Iron, per cent	Producer	Tensile Strength, psi				Yield Strength, psi				Elongation, per cent			
Laboratory....		X	Y	Z	Average	X	Y	Z	Average	X	Y	Z	Average
					ALLOY SC84A								
No. 5714:	A..	47 500	49 700	48 200	48 400	21 600	23 900	22 500	22 700	4.0	4.1	3.8	4.0
0.8........	C..	51 300	52 200	52 000	51 800	26 400	28 100	27 000	27 200	4.3	5.0	4.5	4.6
Average.....					50 100				25 000				4.3
No. 5715:	A..	45 800	46 900	47 400	46 700	20 800	23 200	22 100	22 000	3.1	3.8	3.7	3.5
1.2........	C..	50 600	51 300	50 500	50 800	25 700	27 300	25 700	26 200	4.3	4.6	3.8	4.2
Average.....					48 800				24 100				3.8
No. 5716:	A..	46 200	48 800	46 300	47 800	21 400	23 900	22 400	22 600	2.9	3.5	2.8	3.1
1.6........	C..	48 200	50 500	49 400	49 000	26 000	26 500	26 000	26 200	3.6	3.3	3.1	3.3
Average.....					48 400				24 400				3.2
No. 5717:	A..	46 000	46 200	46 700	46 300	21 700	23 600	23 100	22 800	2.5	2.5	2.6	2.5
2.0........	C..	49 100	49 200	49 600	49 300	26 800	28 200	27 600	27 500	2.7	3.0	2.6	2.8
Average.....					47 800				25 200				2.6
Grand Averages...					48 800				24 700				3.5
					ALLOY SC114A								
Lot 1:	A..	48 100	45 600	45 200	46 300	23 600	23 900	22 700	23 400	3.0	2.5	2.3	2.6
0.8........	B..	53 200	54 000	52 300	53 200	26 000	26 400	25 200	25 900	4.2	4.2	3.4	3.9
	C..	45 400	44 800	46 900	45 700	24 700	24 700	25 200	24 900	2.6	2.6	2.2	2.4
Average.....					48 400				24 700				2.9
Lot 2:	A..	48 400	45 900	45 700	46 700	22 900	22 800	22 400	22 700	2.1	2.6	2.1	2.3
1.2........	B..	51 700	52 100	52 400	52 100	27 800	26 100	25 400	26 400	3.6	3.8	3.2	3.5
	C..	43 700	45 300	45 600	44 900	24 200	24 000	23 600	23 900	2.2	2.9	2.0	2.4
Average.....					47 900				24 300				2.7
Lot 3:	A..	46 900	46 800	45 700	46 500	23 800	24 900	23 400	24 000	1.4	2.0	1.7	1.7
1.6........	B..	53 700	52 600	53 300	53 200	25 800	27 600	26 700	26 700	2.8	2.8	2.4	2.6
	C..	43 200	43 000	43 900	43 400	25 300	25 300	25 100	25 200	1.8	2.0	1.3	1.7
Average.....					47 700				25 300				2.0
Grand Averages...					48 000				24 800				2.5

Co., and the Pitman-Dunn Laboratories of Frankford Arsenal. The bars were tested for ultimate tensile strength, 0.2 per cent offset yield strength, and per cent elongation.

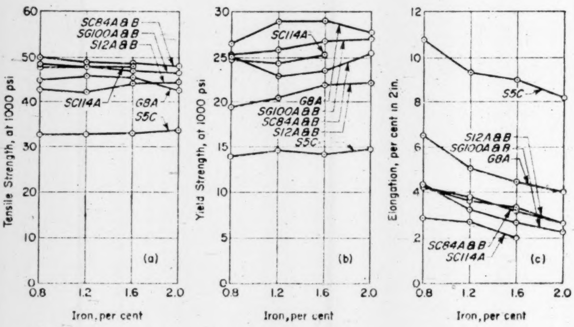
Test Results:

The results of the mechanical tests performed are shown in Table II. The grand averages are plotted in Fig. 2. Each single value in Table

TABLE II.—*Concluded.*

Iron, per cent	Producer	Tensile Strength, psi				Yield Strength, psi				Elongation, per cent			
Laboratory.....		X	Y	Z	Average	X	Y	Z	Average	X	Y	Z	Average
					ALLOY SG100A AND SG100B								
Lot 1:	A..	45 600	45 600	45 500	45 600	22 800	22 800	22 700	22 800	4.3	4.5	4.1	4.3
0.8........	C..	50 400	49 300	49 000	49 600	27 600	27 800	26 700	27 400	4.6	4.1	4.6	4.4
Average.....					47 600				25 100				4.4
Lot 2:.......	A..	46 100	46 000	45 200	45 800	23 900	24 000	24 000	24 000	3.5	3.5	2.8	3.3
1.2.........	C..	49 800	49 300	49 200	49 400	27 400	27 700	27 000	27 400	2.9	3.1	4.0	3.3
Average.....					47 600				25 700				3.3
Lot 3:	A..	43 800	44 100	44 500	44 100	24 600	24 700	24 700	24 700	2.2	2.5	2.0	2.2
1.6.........	C..	50 100	49 300	48 600	49 300	28 700	29 500	28 000	28 700	2.7	3.4	3.3	3.1
Average.....					46 700				26 700				2.7
Lot 4:.......	A..	42 100	42 200	42 100	42 100	24 900	25 800	24 900	25 200	1.7	2.0	1.5	1.7
2.0........	C..	50 900	49 700	49 700	50 100	28 300	29 200	28 400	28 600	2.5	3.1	3.0	2.9
Average.....					46 100				26 900				2.3
Grand Averages...					47 000				26 100				3.2
					ALLOY G8A								
Lot 1:	A..	44 800	44 500	43 000	44 100	26 600	26 700	25 400	26 200	6.9	6.5	5.9	6.4
0.8........	B..	45 800	43 800	46 100	45 200	26 100	26 900	26 900	26 600	7.7	6.0	8.0	7.2
	C..	45 800	45 200	45 900	45 600	29 400	29 100	28 000	28 800	5.5	5.2	6.2	5.6
Average.....					45 000				27 200				6.4
Lot 2:	A..	46 700	45 400	44 900	45 700	28 700	29 700	27 400	28 600	5.7	4.5	4.8	5.0
1.2........	B..	46 600	45 800	46 900	46 400	28 500	29 200	28 600	28 800	6.1	5.0	5.7	5.6
	C..	44 300	44 900	45 500	44 900	30 300	29 200	29 100	29 500	4.2	4.2	5.0	4.4
Average.....					45 700				29 000				5.0
Lot 3:	A..	45 700	44 200	43 300	44 400	29 100	29 700	27 900	28 900	4.0	3.7	3.5	3.7
1.6........	B..	44 900	44 000	45 500	44 800	28 900	29 500	29 200	29 200	4.6	4.0	4.8	4.4
	C..	46 200	45 600	45 500	45 800	29 300	29 300	28 000	28 900	5.2	4.6	5.5	5.1
Average					45 000				29 000				4.4
Lot 4:	A..	46 000	43 400	44 300	44 600	27 800	27 800	26 900	27 500	4.9	4.2	4.9	4.6
2.0........	B..	38 800	38 900	39 800	39 200	27 200	28 000	27 300	27 500	3.0	2.6	3.6	3.6
	C..	44 200	42 400	41 400	42 700	28 100	28 200	29 900	28 700	5.5	4.2	4.3	4.6
Average.....					42 200				27 900				4.0
Grand Averages...					44 500				28 300				5.0

II is the average of 15 test bars and each average in these tables is the average of 45 bars. Producer B did not submit bars of the SC100A and SG100B alloys. For the properties of SC84A and SC84B, in Table II, values for bars produced by producer B have been omitted because these bars exhibited abnormal properties, possibly induced by an artificial aging due to an inadvertent high-temperature storage.



(*a*) Effect upon tensile strength.
(*b*) Effect upon yield strength.
(*c*) Effect upon elongation.

FIG. 2.—Effect of Iron Content in Aluminum Die-Casting Alloys.

The plotted results (Fig. 2) show that, in all cases, the per cent elongation of aluminum die-casting alloys decreases with increasing iron content. The effect of increasing iron content on ultimate tensile strength is variable; in some instances the slope of the curve is positive while, in others, it is negative. Each plotted point, except those for which producer B data are not included, is the result of approximately 135 tests. Over the level of iron contents studied, resultant changes in the ultimate tensile strength range from a decrease of 5 per cent to an increase of 4 per cent. The net change in yield strength for 0.8 to 2.0 per cent iron resulted in an increase for each alloy, the greatest increase being 13 per cent for the S12 alloy.

Serious degradation of ductility, as evidenced by losses in elongation of 38 to 48 per cent, occurs in the S12A, S12B, SC84A, SC84B, and SC114A alloys as the iron content is increased. The S5C alloy suffered a loss of approximately 22 per cent in elongation from 0.8 to 2.0 per cent iron. The loss is not regarded as being serious in this alloy since the alloy still retains a good measure of elongation, even at the 2.0 per cent iron level.

Respectfully submitted by the task group,

D. H. KLEPPINGER (Chairman),
E. V. BLACKMUN,
W. W. BROUGHTON,
D. L. COLWELL,
J. C. FOX,
A. E. MARTIN.

REPORT OF COMMITTEE B-7

ON

LIGHT METALS AND ALLOYS, CAST AND WROUGHT*

Committee B-7 on Light Metals and Alloys, Cast and Wrought, held two meetings during the year: on June 28, 1962, in New York, N. Y., in conjunction with the Annual Meeting of the Society; and on Dec. 5, 1962, in Washington, D. C. The subcommittees and the Advisory Committee met in conjunction with the committee meetings.

The committee consists of 95 members, all of whom are voting members; 34 are classified as producers, 39 as consumers, and 22 as general interest members.

The committee deeply regrets to report the death of E. H. Dix on Jan. 26, 1962, and G. V. Kingsley on Jan. 28, 1962, and R. C. Schubert on Jan. 11, 1962, members of the committee since 1928, 1951, and 1960 respectively.

T. E. Gregory was appointed chairman of Subcommittee IX, Editorial, to succeed F. M. Howell. Appointment of the following representatives to other ASTM committees was made: H. Y. Hunsicker on Subcommittee VII, Electromagnetic Methods, of Committee E-7 on Nondestructive Testing; F. M. Howell to replace H. J. Rowe on Committee E-8 on Nomenclature and Definitions; and J. C. Millson to replace R. F. Hafer on Committee E-12 on Appearance of Materials. W. G. Snyder was appointed to represent the Society on ASA Sectional Committee B96 on Welded Aluminum-Alloy Field-Erected Storage Tanks.

The Advisory Committee of Committee B-7 continued to function as advisor to the American Standards Assn. in its representation of the United States as a participating member of ISO Technical Committee 79 on Light Metals and Their Alloys. The working groups of ISO/TC 79 with United States members are: WG1, Terminology, R. B. Smith; WG3, Magnesium and Its Alloys, A. A. Moore; WG4, Wrought Aluminum Alloys, H. D. Monsch; WG5, Mechanical Characteristics of Castings of Aluminum and Magnesium Alloys, D. L. Colwell and A. A. Moore; WG6, Methods of Mechanical Testing, J. C. Millson; and WG7, Symbols, C. B. Gleason. The United States is not represented on WG2, Unalloyed Aluminum Ingots, and on Subcommittee I, Methods of Chemical Analysis.

At the 1962 meetings of ISO/TC 79 in Paris, France on Nov. 20 to 23, the United States was represented by the Canadian delegation, consisting of J. W. Meier (Head of U. S. delegation), D. C. Sunnucks, and J. H. Snelgrove; and by J. R. Churchill and G. C. Holme.

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

RECOMMENDATIONS AFFECTING STANDARDS

The committee recommends 2 new specifications for publication as tentative, revision of 2 tentative specifications and their continuation as tentative, adoption of 2 tentatives as standard with revisions, revision of 21 standards for immediate adoption, withdrawal of 2 standards, and acceptance of 13 standards by the American Standards Assn. as American Standard. The standards and tentatives affected, together with the revisions recommended, are covered in detail in Appendix I to this report.[1]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Aluminum and Aluminum-Alloy Ingots (D. L. LaVelle, chairman) revised the Specification for Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 179) to add new alloy SC94A, to delete the section on rehearings, and to revise the sections on chemical requirements, sampling, and methods of analysis to agree with current industry practice. The subcommittee recommends that the revised specification be accepted by the American Standards Assn. as American Standard.

Subcommittee II on Aluminum Alloy Castings (D. L. Colwell, chairman) revised the Specification for Aluminum-Base Alloy Sand Castings (B 26) and the Specification for Aluminum-Base Alloy Permanent Mold Castings (B 108) to revise the sections on chemical requirements, methods of analysis, and sampling for analysis and tension testing to agree with current industry practice, and to add to B108 new alloy SC94A and dimensions for the minimum size of tension test specimens cut from castings. The subcommittee recommends that the revised specifications be adopted as standard.

A joint task group of the subcommittee and the Die Casting and Permanent Mold Division of the American Foundrymen's Society is attempting to develop a standard design for a permanent mold for aluminum alloy tension test specimens.

Subcommittee III on Wrought Aluminum and Wrought Aluminum Alloys (H. D. Monsch, chairman) developed the new Specification for Aluminum-Alloy Seamless Condenser and Heat-Exchanger Tubes With Integral Fins and revised the following specifications to change the sections on chemical requirements, sampling, and methods of analysis to agree with current industry practice, to change dimensions for the minimum size of tension test specimens, and to specify that discoloration from heat treatment is not cause for rejection, and additional changes, as shown in Appendix I to this report:[1]

Standard Specifications for:

Aluminum-Alloy Sheet and Plate (B 209 – 62) to add alloys and tempers, thickness tolerances and marking conforming to ASME Pressure Vessel Code requirements, and flatness tolerances for stress-relieved plate.

Aluminum-Alloy Drawn Seamless Tubes (B 210 – 62) to add alloy 5086.

Aluminum-Alloy Bars, Rods, and Wire (B 211 – 62).

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[1] See p. 197.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Aluminum-Alloy Extruded Bars, Rods, and Shapes (B 221 – 62) to add the requirements for extruded tube which are in Specification B 235.

Aluminum-Alloy Drawn Seamless Tubes for Condensers and Heat Exchangers (B 234 – 61).

Aluminum Bars for Electrical Purposes (Bus Bars) (B 236 – 61).

Aluminum-Alloy Pipe (B 241 – 62) to add alloy 5086 and to revise the tensile requirements for 6351-T6.

Aluminum-Alloy Die Forgings (B 247 – 61) to add alloys 5083 and 5456.

Aluminum-Alloy Drawn Seamless Coiled Tubes for Special Purpose Applications (B 307 – 62) to add alloys 3004, 5005, and 5052, and requirements for Type A and Type B tubes which are in Specification B 318.

Aluminum-Alloy Standard Structural Shapes, Rolled or Extruded (B 308 – 62) to revise the tensile requirements for 6351-T6, and to add tolerances for large shapes.

Aluminum-Alloy Round Welded Tubes (B 313 – 61).

Aluminum-Alloy Rivet and Cold Heading Wire and Rods (B 316 – 61).

Aluminum-Alloy Extruded Bar, Rod, Pipe, and Structural Shapes for Electrical Purposes (Bus Conductor) (B 317 – 61).

Aluminum-Alloy Pipe for Gas and Oil Transmission and Distribution Piping Systems (B 345 – 61).

Tentative Specifications for:

Factory Made Wrought Aluminum and Aluminum-Alloy Welding Fittings (B 361 – 61 T).

Aluminum Foil for Capacitors (B 373 – 61 T).

The subcommittee recommends that the Specification for Aluminum-Alloy Extruded Tubes (B 235) be withdrawn and its requirements incorporated in the revision of Specification B 221, that the Specification for Type A and Type B Aluminum-Alloy Drawn Annealed Seamless Coiled Tubes (B 318) be withdrawn and its requirements incorporated in the revision of Specification B 307, and that the revisions of all standard specifications under the jurisdiction of the subcommittee be accepted by the American Standards Assn. as American Standard.

Subcommittee IV on Magnesium and Magnesium Alloys, Cast and Wrought (A. A. Moore, chairman) developed the new Specification for Magnesium-Base Alloy Investment Castings and revised the following specifications, as shown in Appendix I to this report:[1]

Magnesium-Base Alloy Sand Castings (B 80 – 62), and Magnesium-Base Alloy Permanent Mold Castings (B 199 – 62) to specify a specific size tension test specimen and to reference the Reference Radiographs for Inspection of Aluminum and Magnesium Castings (E 155).

Magnesium-Base Alloy Sheet and Plate (B 90 – 61) to increase the mechanical property limits and add additional thicknesses for HM21A-T8, to revise dimensions for the minimum size of tension test specimens, and to specify the location from which tension test specimens shall be taken.

Magnesium-Base Alloy Forgings (B 91 – 60) to revise dimensions for the minimum size of tension test specimens.

Magnesium-Base Alloy Bars, Rods, and Shapes (B 107 – 61) to revise dimensions for the minimum size of tension test specimens and to specify the location from which tension test specimens shall be taken.

Magnesium-Base Alloy Extruded Tubes (B 217 – 60) to add mechanical property limits for additional sizes of tube, to revise dimensions for the minimum size of tension test specimens, and to specify the location from which tension test specimens shall be taken.

Subcommittee V on Testing Light Metals (J. C. Millson, chairman) is cooperating with Subcommittee 4 on Tension Testing of Committee E-1 on Methods of Testing in establishing equivalence of subsize flat tension test specimens with the standard $\frac{1}{2}$-in. wide standard flat specimen shown in Fig. 7 of the Methods of Tension Testing of Metallic Materials (E 8), and establishing standard sizes for subsize flat specimens; and in resolving discrepancies between Method E 8 and Federal Test Method Standard No. 151, and making these standards more specific concerning specimens for testing light metals. The subcommittee is also reviewing sampling requirements in ASTM and Government specifications for light metals in preparation for making recommendations for achieving greater uniformity in the sampling requirements.

Subcommittee VII on Codification of Light Metals and Alloys, Cast and Wrought (C. B. Gleason, chairman) compiled the list of alloy designations assigned in conformance with the Recommended Practice for Codification of Light Metals and Alloys, Cast and Wrought (B 275-61), and the ASTM specifications in which the alloys appear, which is Appendix II to this report[3]; and assigned a number of new designations during the year.

Subcommittee VIII on Atmospheric Exposure Tests (L. H. Adam, chairman) has test specimens of 27 aluminum and 8 magnesium commercial alloys, cast and wrought, at five ASTM atmospheric exposure test sites: on top of the Port Authority Building, New York, N. Y., for industrial atmospheric conditions; at State College, Pa., for rural; at Kure Beach, N. C., for Atlantic Coast marine; at Point Reyes, Calif., for Pacific Coast marine; and at Freeport, Tex., for Gulf Coast marine atmospheric conditions. Data have been published in ASTM *Proceedings*[4] for specimens exposed six months, one year, three years, and five years. Data on specimens exposed for 10 yr are now being generated for a future report to the Society.

Subcommittee IX, Editorial (T. E. Gregory, chairman) is preparing Recommendations on Form of ASTM Committee B-7 Specifications to supplement Recommendations on Form of ASTM Standards, April, 1961, which have been approved as a guide, insofar as practical, for writing and revising Committee B-7 specifications. Standardization of section titles, sequence, wording, form, titles, and headings of tables is being attempted.

Special Subcommittee on Light Metals for Nuclear Applications (W. G. Groth, chairman) has kept informed about the special requirements of the nuclear industry to determine whether new or revised specifications are required for light metals for nuclear applications, and recommended to Subcommittee III that specifications be written for wrought aluminum - aluminum oxide products made from aluminum powder.

This report has been submitted to letter ballot of the committee, which consists of 94 members; 80 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. A. HARRIS,
Chairman.

R. B. SMITH,
Secretary

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[3] See p. 210.

[4] Data for 6-month and 1-yr exposures: L. H. Adam, "Atmospheric Exposure of Aluminum and Magnesium Sand and Permanent Mold Castings," *Proceedings*, Am. Soc. Testing Mats., Vol. 54, p. 270 (1954); L. H. Adam and M. Dougherty, "Atmospheric Exposure of Wrought Aluminum and Magnesium Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 55, p. 284 (1955).

Data for 3-yr exposures: L. H. Adam and M. Dougherty, "Atmospheric Exposure of Aluminum and Magnesium Sand and Permanent Mold Castings," *Proceedings*, Am. Soc. Testing Mats., Vol. 58, p. 194 (1958); L. H. Adam and M. Dougherty, "Atmospheric Exposure of Wrought Aluminum and Magnesium Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 59, p. 289 (1959).

Data for 5-yr exposures: L. H. Adam and M. Dougherty, "Atmospheric Exposure of Aluminum and Magnesium Sand and Permanent Mold Castings," *Proceedings*, Am. Soc. Testing Mats., Vol. 60, p. 186 (1960); L. H. Adam and M. Dougherty, "Atmospheric Exposure of Wrought Aluminum and Magnesium Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 61, p. 302 (1961). L. H. Adam and M. Dougherty, "Atmospheric Exposure of Aluminum Alloy Sand Casting G10A-T4 (Commercial 220)," *Proceedings*, Am. Soc. Testing Mats., Vol. 62, p. 265 (1962).

APPENDIX I

PROPOSED RECOMMENDATIONS AFFECTING STANDARDS ON LIGHT METALS AND ALLOYS, CAST AND WROUGHT

Committee B-7 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting. All of the existing tentatives and standards referred to in this report appear in their present form in the 1961 Book of ASTM Standards, Part 2, or the 1962 Supplement thereto.

New Tentatives

as appended hereto.[1]

Tentative Specifications for:

Aluminum-Alloy Seamless Condenser and Heat-Exchanger Tubes with Integral Fins, and

Magnesium-Base Alloy Investment Castings.

Revision of Tentatives

Tentative Specification for Factory Made Wrought Aluminum and Aluminum-Alloy Welding Fittings (B 361 – 61 T):

Table I.—Change all references to Specification B 235 to read "B 221." In Footnote *d* delete B 235 and change the title of B 221 to read "Aluminum-Alloy Extruded Bars, Rods, Shapes, and Tubes." Change Footnote *b* to read: "[b] These alloy designations were established in accordance with the American Standard Alloy and Temper Designation Systems for Wrought Aluminum (ASA No.: H35.1), except for the letter symbols 'WP'."

Section 5.—Add the following to Paragraph (*a*):

Conformance shall be determined by the manufacturer by analyzing samples taken at the time the ingots are poured, or samples taken from the finished or semi-finished product. If the manufacturer of the fittings or the manufacturer of the material from which the fittings are made has determined the chemical composition of the material during the course of manufacture, sampling and analysis of the finished product shall not be required.

Section 8.—Change Paragraph (*b*) to read as follows including the new Note 3:

(*b*) The fittings shall be free from injurious defects and shall have a workmanlike finish. Discoloration characteristic of proper solution heat treatment shall not be cause for rejection.

NOTE 3.—Principles of proper solution heat treatment are described in Military Specification MIL-H-6088, Heat Treatment of Aluminum Alloys.

Section 9.—Change the heading to "Sampling for Determination of Chemical Composition" and change to read as follows:

9. (*a*) The number of samples taken for determination of chemical composition shall be as follows:

(*1*) When samples are taken at the time the ingots are poured, at least one sample shall be taken for each group of ingots poured simultaneously from the same source of molten metal.

(*2*) When samples are taken from the finished or semi-finished product, a sample shall be taken to represent each 4000 lb or fraction thereof of material in the shipment, except that not more than one sample shall be required per piece.

(*b*) Samples for determination of chemical composition shall be taken in accordance with one of the following methods:

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[1] The new tentatives appear in the 1963 Supplement to *Book of ASTM Standards*, Part 2.

(*1*) *Samples for Chemical Analysis.*—Method of sampling wrought nonferrous metals and alloys shall be by drilling, sawing, milling, turning, clipping, etc., a representative piece or pieces to obtain a weight of prepared sample not less than 75 g. (See the Method of Sampling Wrought Nonferrous Metals (ASTM Designation: E 55)).

(*2*) *Samples for Spectrochemical and Other Methods of Analysis.*—Method of sampling wrought products shall be suitable for the form of material being analyzed and the type of analytical method used.

NOTE 4.—The analysis of clad material in the finished state is a difficult matter because of the relatively thin layer of cladding present and the effect of diffusion between the core and cladding during thermal treatment. A reasonably satisfactory determination of the core composition can be made if the cladding is substantially removed from the sample before analysis. Determination of the cladding analysis is considerably more difficult not only because of its thinness, but also because diffusion causes an indeterminate change in the composition. That the correct cladding alloy has been used, however, can usually be determined reasonably well by a combination of metallographic examination and spectrochemical analysis of the surface at several widely separated points.

Section 10.—Change Paragraph (*a*) to read: "(*a*) The determination of chemical composition shall be made in accordance with suitable chemical, spectrochemical, or other methods." Reference a footnote to chemical methods to read: "1960 Book of ASTM Methods for Chemical Analysis of Metals," and to spectrochemical methods to read: "Methods for Emission Spectrochemical Analysis (1960)."

Section 12.—Renumber Note 4 as Note 5.

Section 15.—Delete this section.

Tentative Specification for Aluminum Foil for Capacitors (B 373 – 61 T):

Section 3.—Change to read as follows under the heading "Chemical Composition:"

3. The foil shall conform to the chemical composition limits prescribed in Table I. Conformance shall be determined by the manufacturer by analyzing samples taken at the time the ingots are poured, or samples taken from the finished or semi-finished product. If the manufacturer of the foil or the manufacturer of the material from which the foil is produced has determined the chemical composition of the material during the course of manufacture, sampling and analysis of the finished product shall not be required.

Table I.—Change Footnote *a* to read: "These alloy designations were established in accordance with the American Standard Alloy and Temper Designation Systems for Wrought Aluminum (ASA No.: H35.1)."

Section 12.—Change the heading to "Sampling for Determination of Chemical Composition" and change to read as follows:

12. (*a*) The number of samples taken for determination of chemical composition shall be as follows:

(*1*) When samples are taken at the time the ingots are poured, at least one sample shall be taken for each group of ingots poured simultaneously from the same source of molten metal.

(*2*) When samples are taken from the finished or semi-finished product, a sample shall be taken to represent each 1000 lb or fraction thereof of material in the shipment, except that not more than one sample shall be required per piece.

(*b*) Samples for determination of chemical composition shall be taken in accordance with one of the following methods:

(*1*) *Samples for Chemical Analysis.*—Method of sampling wrought nonferrous metals and alloys shall be by drilling, sawing, milling, turning, clipping, etc., a representative piece or pieces to obtain a weight of prepared sample not less than 75 g. (See the Method of Sampling Wrought Nonferrous Metals (ASTM Designation: E 55)).

(*2*) *Samples for Spectrochemical and Other Methods of Analysis.*—Method of sampling wrought products shall be suitable for the form of material being analyzed and the type of analytical method used.

Section 14.—Revise Paragraph (*a*) to read as indicated above for Section 10 of Specification B 361.

Adoption of Tentatives as Standard With Revision

Tentative Specification for Aluminum-Base Alloy Sand Castings (B 26 – 62 T):

Section 4.—Change to read as follows:

4. The castings shall conform to the chemical composition limits prescribed in Table I. Conformance shall be determined by the manufacturer by analyzing samples taken at the time the castings are poured, or samples taken from castings or tension test specimens representative of the castings. If the manufacturer has determined the chemical composition of the material during the course of manufacture, he shall not be required to sample and analyze the finished product.

Section 5.—Change the heading to "Sampling for Determination of Chemical Composition" and change to read as follows:

5. (*a*) A sample for determination of chemical composition shall be taken to represent the following:

(*1*) not more than 1000 lb of clean castings poured from a batch-type furnace; or

(*2*) the castings poured from one continuous furnace in not more than 8 consecutive hr.

(*b*) Samples for determination of chemical composition shall be taken in accordance with one of the following methods:

(*1*) *Samples for Chemical Analysis.*—Samples for chemical analysis shall be taken by sawing, drilling, or milling the casting or test specimens in such a manner as to be representative of the material. The weight of a prepared sample shall be not less than 75 g. (See Methods of Sampling Nonferrous Alloys in Cast Form for Determination of Chemical Composition (ASTM Designation: E 88)).

(*2*) *Samples for Spectrochemical and Other Methods of Analysis.*—Samples for spectrochemical analysis or other methods of analysis shall be suitable for the form of material being analyzed and the type of analytical method used.

Section 6.—Change the heading to "Methods for Determination of Chemical Composition" and change to read: "The determination of chemical composition shall be made in accordance with suitable chemical, spectrochemical, or other methods." Reference a footnote to chemical methods to read: "1960 Book of ASTM Methods for Chemical Analysis of Metals," and to spectrochemical methods to read: "Methods for Emission Spectrochemical Analysis (1960)."

Section 9.—Change Paragraph (*a*) to read as follows:

9. (*a*) Unless otherwise agreed upon by the purchaser and manufacturer, two tension test specimens shall be separately cast and tested to represent the following:

(*1*) not more than 1000 lb of clean castings poured from a batch-type furnace; or

(*2*) the castings poured from one continuous furnace in not more than 8 consecutive hr.

Tentative Specification for Aluminum-Base Alloy Permanent Mold Castings (B 108 – 62 T):

Sections 4, 5, and 6.—Change as indicated above for the Sections 4, 5, and 6 in Specification B 26.

Section 8.—Change Paragraph (*b*) to read as follows:

(*b*) When properties of SC51B and SG70B alloy castings are to be determined, tension test specimens shall be cut from the locations designated on the drawing, unless otherwise negotiated. If no locations are designated, one or more specimens shall be taken to include locations having significant variation in casting thickness, except that specimens shall not be taken from areas directly under risers. The tension test specimens shall be the standard ½-in. diameter specimen shown in Fig. 8 of the Methods of Tension Testing of Metallic Materials (ASTM Designation: E 8) or a round specimen of smaller size proportional to the standard specimen. In no case shall the dimensions of the smallest specimen be less than the following:

Diameter of reduced section, in.	¼
Length of reduced section, in.	1¼
Radius of fillet, in.	3/16
Diameter of end section, in.	⅜
Over-all length, in.:	
With shouldered ends	2⅜
With threaded ends	3
With plain cylindrical ends	4

When necessary, a rectangular specimen may be used proportional to that shown in Fig. 7 of

Methods E 8, but in no case shall its dimensions be less than the following:

Width of reduced section, in.	¼
Length of reduced section, in.	1¼
Radius of fillet, in.	¼
Over-all length, in.	4

The specified elongation values shall not apply to tests of rectangular specimens.

Section 9.—Revise as indicated above for Section 9 in Specification B 26.

Table I.—Add alloy SC94A with the following chemical requirements:

Aluminum, per cent	remainder
Copper, per cent	3.0 – 4.0
Iron, per cent	1.0
Silicon, per cent	8.0 – 10.0
Manganese, per cent	0.50
Magnesium, per cent	0.05 – 0.50
Zinc, per cent	1.0
Titanium, per cent	0.25
Nickel, per cent	0.50
Other Elements, total, per cent	0.50

Table II.—Add alloy SC94A with the following tensile requirements and reference to Footnote *c* in the elongation column:

Condition	Tensile Strength, min, psi
F (As cast)	28 000
T5 (Aged)	30 000
T6 (Solution treated and aged)	35 000
T7 (Solution treated and overaged)	31 000

Table IV.—Add alloy SC94A with the following properties and characteristics:

Approximate melting range, deg Fahr	960 – 1085
Foundry Characteristics:	
Resistance to hot cracking	2
Pressure tightness	2
Fluidity	2
Solidification shrinkage tendency	3
Normally heat-treated	No
Other Characteristics:	
Resistance to corrosion	3
Machining	3
Polishing	3
Electroplating	3
Anodizing (appearance)	5
Chemical oxidation coating	3
Strength at elevated temperature	3
Suitability for welding	2
Suitability for brazing	No

Revision of Standards, Immediate Adoption

(requires nine-tenths affirmative vote)

Standard Specification for Magnesium-Base Alloy Sand Castings (B 80 – 62):

Section 9.—Change the first sentence in Paragraph (*a*) to read as follows:

> The tension test specimens shall be separately cast in green sand and shall be cast to size in accordance with the dimensions shown for the standard ½-in. diameter test specimen shown in Fig. 8 of the Methods of Tension Testing of Metallic Materials (ASTM Designation: E 8) except that the radii at the ends of the reduced section shall be not less than ½ in.

Section 15.—In Paragraph (*c*) change designation E 98 to read E 155.

Standard Specification for Magnesium-Base Alloy Sheet and Plate (B 90 – 61):

Section 8.—Change to read as follows:

> 8. (*a*) For sheet and plate less than 0.500 in. in thickness the tension test specimen shown in Fig. 7, or for plate 0.500 in. or over those in Fig. 8 of the Methods of Tension Testing of Metallic Materials (ASTM Designation: E 8) shall be used. If it is necessary to use specimens smaller than the standard specimens, they shall have dimensions proportional to those of Fig. 8 but not less than the following dimensions: reduced section, ¼ in. diameter by 1 in. gage length; grip ends, ⅜ in. diameter; total length, 2⅜ in. with shouldered ends, 3 in. with threaded ends, and 4 in. if tested with plain cylindrical ends. If material less than ¾ in. in width is tested in full section because the specimens in Fig. 8 cannot be used, the elongation shall not be determined.
>
> (*b*) Tension test specimens shall be taken parallel to the direction of rolling. The specimen shall be taken midway between the two plate surfaces for plate in thicknesses of 0.500 in. through 1.500 in., and midway between the center and the surface of plate over 1.500 in. in thickness.

Table II.—Revise the tensile requirements for HM21A-T8 to read as shown in accompanying Table I.

Standard Specification for Magnesium-Base Alloy Forgings (B 91 – 60):

Section. 8—Change the last sentence of this section to read as follows:

If it is necessary to use specimens smaller than the standard specimens, they shall have dimensions proportional to those of Fig. 8 of Methods E 8 but not less than the following dimensions: reduced section, $\frac{1}{4}$ in. diameter by 1 in. gage length; grip ends, $\frac{3}{8}$ in. diameter; total length, $2\frac{3}{8}$ in. with shouldered ends, 3 in. with threaded ends, and 4 in. if tested with plain cylindrical ends.

TABLE I.—TENSILE REQUIREMENTS.
(Revision in Table II of Specification B 90)

Alloy and Condition	Thickness, in.	Tensile Strength, min, psi	Yield Strength (0.2 per cent offset), min, psi	Elongation in 2 in., min, per cent
HM21A-T8............	0.016 – 0.250	33 000	18 000	6
	0.251 – 0.500	32 000	21 000	6
	0.501 – 1.000	30 000	21 000	6
	1.001 – 3.000	30 000	21 000	6

Standard Specification for Magnesium-Base Alloy Bars, Rods, and Shapes (B 107 – 61):

Section. 8—Change the last sentence in Paragraph (*a*) to read as indicated above for Section 8 of Specification B 91. Change Paragraph (*b*) to read as follows:

(*b*) Tension test specimens shall be taken parallel to the direction of extrusion. For material $1\frac{1}{2}$ in. and less in diameter or thickness when not tested in full section, the specimen shall be taken from the center of the section. For material greater than $1\frac{1}{2}$ in. in diameter or thickness, the specimen shall be taken from midway between the center and the surface. For extruded shapes, the specimen shall be taken from the predominant part of the shape and the location within the predominant part be as given above.

Standard Specification for Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 179 – 62):

Sections 5, 6, 7, and 8.—Delete and replace with the following, renumbering succeeding sections accordingly:

Chemical Composition Requirements

5. The ingots shall conform to the chemical composition limits prescribed in Table I. Conformance shall be determined by the manufacturer by analyzing samples taken at the time the ingots are poured, or samples taken from the ingots. If the manufacturer has determined the chemical composition of the material during the course of manufacture, he shall not be required to sample and analyze the ingots.

Sampling for Determination of Chemical Composition

6. (*a*) The number of samples taken for determination of chemical composition shall be as follows:

(*1*) When samples are taken at the time the ingots are poured, at least one sample shall be taken for each group of ingots poured from the same source of molten metal.

(*2*) If the ingots are shipped in carload lots of the same alloy, not less than five ingots shall be taken at random from the carload for sampling. If the shipment is in less than carload lots, one sample ingot shall be taken for each 6000 lb or fraction thereof. When it is deemed necessary, a sample may be taken from each melt of 500 lb or more of the alloy.

(*b*) Samples for determination of chemical composition shall be taken in accordance with one of the following methods:

(*1*) *Samples for Chemical Analysis.*—Samples for chemical analysis shall be taken by sawing, drilling, or milling ingots or test specimens in such a manner as to be representative of the material. The weight of a prepared sample shall be not less than 75 g (see Method of Sampling Nonferrous Metals and Alloys in Cast Form for Determination of Chemical Composition (ASTM Designation: E 88)).

(2) *Samples for Spectrochemical and Other Methods of Analysis.*—Samples for spectrochemical and other methods of analysis shall be suitable for the form of material being analyzed and the type of analytical method used.

Methods for Determination of Chemical Composition

7. The determination of chemical composition shall be made in accordance with suitable chemical, spectrochemical, or other methods. (Reference a footnote to chemical methods to read: "1960 Book of ASTM Methods for Chemical Analysis of Metals;" and to spectrochemical methods to read "1960 Book of ASTM Methods for Emission Spectrochemical Analysis").

Section 17.—Delete the section.

Table I.—Add alloy SC94A with the following chemical requirements, and indicate the principal use as permanent mold cast, P:

Aluminum, per cent	remainder
Copper, per cent	3.0 – 4.0
Iron, per cent	0.9
Silicon, per cent	8.0 – 10.0
Manganese, per cent	0.50
Magnesium, per cent	0.10 – 0.50
Zinc, per cent	1.0
Titanium, per cent	0.25
Nickel, per cent	0.50
Other Elements, total, per cent	0.50

Standard Specification for Magnesium-Base Alloy Permanent Mold Castings (B 199 – 62):

Section 9.—Change the first sentence in Paragraph (*a*) to read:

The tension test specimens shall be separately cast in permanent molds and shall be cast to size in accordance with the dimensions shown for the standard ½-in. diameter test specimen shown in Fig. 8 of the Methods of Tension Testing of Metallic Materials (ASTM Designation: E 8) except that the radii at the ends of the reduced section shall be not less than ½ in.

Section 15.—Change the last sentence in Paragraph (*c*) to read: "Unless specific reference is made to the degree of discontinuity by reference to Reference Radiographs for Inspection of Aluminum and Magnesium Castings (ASTM Designation: E 155) acceptance standards shall be agreed upon by the purchaser and the manufacturer."

Standard Specification for Aluminum-Alloy Sheet and Plate (B 209 – 62):

Revise as appended hereto.[2]

Standard Specification for Aluminum-Alloy Drawn Seamless Tubes (B 210 – 62):

Table I.—Change Footnote *a* to read as indicated above for Footnote *a* in Table I of Specification B 373, and add alloy 5086 with the following chemical requirements:

Aluminum, per cent	remainder
Copper, per cent	0.10
Iron, per cent	0.50
Silicon, per cent	0.40
Manganese, per cent	0.20 – 0.7
Magnesium, per cent	3.5 – 4.5
Zinc, per cent	0.25
Chromium, per cent	0.05 – 0.25
Titanium, per cent	0.15
Other Elements:	
Each, per cent	0.05
Total, per cent	0.15

Table II.—Add alloy 5086 with the following tensile requirements:

Temper	0[a]
Wall Thickness, in.	0.018 – 0.450
Tensile Strength, min. psi	35 000
Yield Strength, min, psi	14 000
Elongation, min, per cent:	
Cut-out Specimen	14

Section 4.—Change to read as follows:

4. The tubes shall conform to the chemical composition limits prescribed in Table I. Conformance shall be determined by the manufacturer by analyzing samples taken at the time the ingots are poured, or samples taken from the finished or semi-finished product. If the manufacturer has determined the chemical composition of the material during the course of manufacture, he shall not be required to sample and analyze the finished product.

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[2] The revised standard appears in the 1963 Supplement to *Book of ASTM Standards*, Part 2.

Section 5.—Change the heading to "Sampling for Determination of Chemical Composition" and change as indicated above for Section 9 of Specification B 361, except delete the number from the note.

Section 6.—Change the heading to "Methods for Determination of Chemical Composition" and change as indicated above for Section 10 of Specification B 361.

Section 8.—Change specimen dimensions to read as follows:

Diameter of reduced section, in.	¼
Length of reduced section, in.	1¼
Radius of fillet, in.	3/16
Diameter of end section, in.	⅜
Over-all length, in.:	
With shouldered ends	2⅜
With threaded ends	3
With plain cylindrical ends	4

Table III.—Add reference to alloy 5086 in Columns 2 and 3.

Table IV.—Add reference to alloy 5086 in Columns 2 and 3.

Section 14.—Add the following sentence and note: "Discoloration characteristic of proper solution heat treatment shall not be cause for rejection. NOTE.—Principles of proper solution heat treatment are described in Military Specification MIL-H-6088, Heat Treatment of Aluminum Alloys."

Section 16.—Delete from the first sentence of Paragraph (*a*): "the latter to be in accordance with the ASA and ASTM codification systems (ASA No. H35.1-1957 and ASTM Designation: B 296)."

Standard Specification for Aluminum-Alloy Bars, Rods, and Wire (B 211 – 62):

Table I.—Change Footnote *a* to read as indicated above for Footnote *a* in Table **I** of Specification B 373.

Section 4.—Change to read as follows:

4. The bars, rods, and wire shall conform to the chemical composition limits prescribed in Table I. Conformance shall be determined by the manufacturer by analyzing samples taken at the time the ingots are poured, or samples taken from the finished or semi-finished product. If the manufacturer has determined the chemical composition of the material during the course of manufacture, he shall not be required to sample and analyze the finished product.

Section 5.—Change the heading to "Sampling for Determination of Chemical Composition" and change as indicated above for Section 9 of Specification B 361, except number the note as "Note 2."

Section 6.—Change the heading to "Methods for Determination of Chemical Composition" and change as indicated above for Section 10 of Specification B 361.

Section 8.—Change specimen dimensions in Paragraph (*a*) as indicated above for Section 8 of Specification B 210.

Section 14.—Change as indicated above for Section 14 of Specification B 210 except number the note as note 4.

Section 15.—Change as indicated above for Section 16 of Specification B 210.

Section 16.—Delete from the first sentence of Paragraph (*b*): "the latter to be in accordance with ASA and ASTM codification systems (ASA No. H35.1-1957 and ASTM Designation: B296)."

Standard Specification for Magnesium-Base Alloy Extruded Tubes (B 217 – 60):

Section 8.—Change to read as follows:

8. Tension test specimens of tubes shall be taken in the longitudinal direction. Tubes 2 in. or less in diameter shall be tested in full section unless the limits of the testing machine preclude the use of such a specimen. For tubes over 2 in. in diameter, or in those cases where a full-section specimen cannot be used, it is permissible to use specimens of the types shown in Fig. 8 and Fig. 13 (Specimen No. 1) of the Methods of Tension Testing of Metallic Materials (ASTM Designation: E 8). If it is necessary to use specimens smaller than the standard

specimens shown in Methods E 8, they shall have dimensions proportional to those of Fig. 8 of Methods E 8 but not less than the following dimensions: reduced section, $\frac{1}{4}$ in. diameter by 1 in. gage length; grip ends, $\frac{3}{8}$ in. diameter; total length, $2\frac{3}{8}$ in. with shouldered ends, 3 in. with threaded ends, and 4 in. if tested with plain cylindrical ends.

Table II.—Revise to read as shown in accompanying Table II.[3] Retain present Footnotes *a* and *b* and delete Footnote *c*.

Standard Specification for Aluminum-Alloy Extruded Bars, Rods, and Shapes (B 221 - 62):

Revise as appended hereto.[2]

Standard Specification for Aluminum-Alloy Drawn Seamless Tubes for Condensers and Heat Exchangers (B 234 - 61):

Sections 4, 5, and 6.—Change as indicated above for the same sections in Specification B 210.

Table I.—Change Footnote *a* to read as indicated above for Footnote *a* in Table I of Specification B 373.

Section 8.—Change the specimen dimensions as indicated above for Section 8 of Specification B 210.

Section 16.—Change Paragraph (*a*) as indicated above for Section 14 of Specification B 210.

Section 17.—Change as indicated above for Section 16 of Specification B 210.

Section 18.—Change as indicated above for Section 16 of Specification B 211.

Standard Specification for Aluminum Bars for Electrical Purposes (Bus Bars) (B 236 - 61):

Section 16.—Change as indicated above for Section 16 of Specification B 210.

Standard Specification for Aluminum-Alloy Pipe (B 241 - 62):

Table I.—Add alloy 5086 with the chemical requirements indicated above for Table I Specification B 210, and change Footnote *a* as indicated above for Footnote *a* in Table I of Specification B 373.

Table II.—Add alloy 5086-0 and revise alloy 6351-T 6 as follows:

	5086-0	6351-T6	
Pipe Size, in.	All	All	All
Wall Thickness, in.	All	Up through 0.124	0.125 - 0.749
Ultimate Strength, min, psi	35 000	42 000	42 000
Yield Strength, min, psi	14 000	37 000	37 000
Elongation, min, per cent	14	8	10

Section 4.—Change to read as follows:

> 4. The pipe shall conform to the chemical composition limits prescribed in Table I. Conformance shall be determined by the manufacturer by analyzing samples taken at the time the ingots are poured, or samples taken from the finished or semi-finished product. If the manufacturer has determined the chemical composition of the material during the course of manufacture, he shall not be required to sample and analyze the finished product.

Section 5.—Change the heading to "Sampling for Determination of Chemical Composition" and change as indicated above for Section 9 of Specification B 361, except exclude the note.

Section 6.—Change the title to "Methods for Determination of Chemical Composition" and change as indicated above for Section 10 of Specification B 361.

Table III.—In Footnote *c* add alloy 5086 between alloys 5083 and 5154.

Section 8.—Change specimen dimensions as indicated above for Section 8 of Specification B 210.

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[3] This table is not included in this report but appears in the revised specification; see 1963 Supplement to *Book of ASTM Standards*, Part 2.

Section 13.—Change Paragraph (*a*) as indicated above for Section 14 of Specification B 210, except number the note as note 3.

Section 14.—Change as indicated above for Section 16 of Specification B 210.

Section 15.—Change as indicated above for Section 16 of Specification B 211.

Standard Specification for Aluminum-Alloy Die Forgings (B 247 – 61):

Table I.—Change Footnote *a* to read as indicated above for Footnote *a* in Table I of Specification B 373. Add alloys 5083 and 5456, change Footnotes *b* and *c*, and add a new footnote *e* to read as follows:

	5083	5456
Aluminum, per cent	remainder	remainder
Copper, per cent	0.10	0.10
Iron, per cent	0.40	[e]
Silicon, per cent	0.40	[e]
Manganese, per cent	0.30 – 1.0	0.50 – 1.0
Magnesium, per cent	4.0 – 4.9	4.7 – 5.5
Zinc, per cent	0.25	0.25
Chromium, per cent	0.05 – 0.25	0.05 – 0.20
Titanium, per cent	0.15	0.20
Other Elements:		
Each, per cent	0.05	0.05
Total, per cent	0.15	0.15

[b] The permissible maximum amount of iron plus silicon is 1.0 per cent.

[c] 99.0 per cent minimum aluminum by difference.

[e] The permissible maximum amount of iron plus silicon is 0.40 per cent.

Table II.—Add alloys 5083-0 and 5456-0 with the following tensile requirements:

	5083-0	5456-0
Tensile strength, min, psi	38 000[c]	40 000[c]
Yield strength, min, psi	16 000[c]	18 000[c]
Elongation, min, per cent	16[c]	16[c]

Section 4.—Change to read as follows:

4. The forgings shall conform to the chemical composition limits prescribed in Table I. Conformance shall be determined by the manufacturer by analyzing samples taken at the time the ingots are poured, or samples taken from the finished or semi-finished product. If the manufacturer has determined the chemical composition of the material during the course of manufacture, he shall not be required to sample and analyze the finished product.

Section 5.—Change the heading to "Sampling for Determination of Chemical Composition" and change to read as follows:

5. (*a*) A sample for determination of chemical composition shall be taken to represent:

(*1*) not more than 2000 lb of forgings when each forging weighs 5 lb or less; or

(*2*) not more than 6000 lb of forgings when the forgings weigh more than 5 lb each.

(*b*) Samples for determination of chemical composition shall be taken in accordance with one of the following methods:

(*1*) *Samples for Chemical Analysis.*—Method of sampling wrought nonferrous metals and alloys shall be by drilling, sawing, milling, turning, clipping, etc., a representative piece or pieces to obtain a weight of prepared sample not less than 75 g. (See the Method of Sampling Wrought Nonferrous Metals (ASTM Designation: E 55)).

(*2*) *Samples for Spectrochemical and Other Methods of Analysis.*—Method of sampling wrought products shall be suitable for the form of material being analyzed and the type of analytical method used.

Section 6.—Change the heading to "Methods for Determination of Chemical Composition" and change as indicated above for Section 10 of Specification B 361.

Section 9.—Change specimen dimensions as indicated above for Section 8 of Specification B 210.

Section 14.—Change as indicated above for Section 14 of Specification B 210.

Section 15.—Change as indicated above for Section 16 of Specification B 211.

Standard Specification for Aluminum-Alloy Drawn Seamless Coiled Tubes for Special Purpose Applications (B 307 – 62):

Section 1.—Change to read as follows:

1. This specification covers aluminum-alloy, drawn, seamless round tubes in coils (Note) suitable for special engineering purposes such as instrument lines, oil lines, refrigeration service, etc., designated as shown in Table I. The tubes designated types A and B in Table II are specific sizes for gas, air, and liquid lines in accordance with the American Gas Association coding procedure.

Retain the note but change the reference to Specification B 235 to read "Specification for Aluminum-Alloy Extruded Bars, Rods, Shapes, and Tubes (ASTM Designation: B 221)."

New Footnote.—Add a new Footnote to read: "This revision includes incorporation of the appropriate requirements of the Specification for Type A and Type B Aluminum-Alloy Drawn Annealed Seamless Coiled Tubes (B 318) which has accordingly been discontinued."

Section 2.—Add the following to Item (6): "or outside diameter and Type (A or B) (Table II)."

Sections 4, 5 and 6.—Change as indicated above for the same sections in Specification B 210.

Section 7.—Change the reference to Table II to read "Table III."

Section 8.—Change to read as follows:

8. Tension test specimens shall be of the full section of the tube unless the limitations of the testing machine preclude the use of such a specimen. Specimen No. 1 of Fig. 13 of the Methods of Tension Testing of Metallic Materials (ASTM Designation: E 8) may be used in those cases when a full-section specimen cannot be used.

Section 11.—Change the reference to Table II to read "Table III."

Section 13.—Change the tabular material to read as follows and retain the note:

Nominal Outside Diameter, in.	Expansion of Outside Diameter, per cent
Up through 0.750	40
0.751 and over	30

Section 15(b).—Change reference to Clad M1A to read "Alclad 3003" in the tabular material.

Section 16.—In Paragraph (*a*) change the tabular material to read as follows, retaining the present footnote:

Specified Outside Diameter, in.	Allowable Deviation of Mean Diameter[a] from Specified Diameter, plus and minus, in.
0.125 – 0.500	0.003
0.501 – 1.000	0.004
1.001 – 2.000	0.005

In Paragraph (*b*) change the reference to Table III to read "Table IV."

Section 18.—Change as indicated above for Section 16 of Specification B 211.

Table I.—Change Footnote *a* as indicated above for Footnote *a* in Table I of Specification B 373. Add a new Footnote *e* and alloys 3004, 5005, and 5052 to read as follows:

	3004	5005	5052
Aluminum, per cent	remainder	remainder	remainder
Copper, per cent	0.25	0.20	0.10
Iron, per cent	0.7	0.7	[e]
Silicon, per cent	0.30	0.40	[e]
Manganese, per cent	1.0 – 1.5	0.20	0.10
Magnesium, per cent	0.8 – 1.3	0.50 – 1.1	2.2 – 2.8
Zinc, per cent	0.25	0.25	0.10
Chromium, per cent	...	0.10	0.15 – 0.35
Other Elements:			
Each, per cent	0.05	0.05	0.05
Total, per cent	0.15	0.15	0.15

[e] Iron plus silicon shall not exceed 0.45 per cent.

New Table.—Add new Table II to read as shown in accompanying Table III.[3]

Table II.—Renumber as Table III and add alloys 3004-0, 5005-0, and 5052-0 with the following tensile requirements:

	3004-0	5005-0	5052-0
Thickness, in.	0.010 – 0.109	0.010 – 0.109	0.010 – 0.109
Tensile strength:			
min, psi	23 000	14 000	25 000
max, psi	29 000	20 000	35 000

Table III.—Renumber as Table IV and add a new wall thickness of "0.084 to 0.109" in the first column, with a tolerance of "0.005" in the second column.

Standard Specifications for Aluminum-Alloy Standard Structural Shapes, Rolled or Extruded (B 308 – 62):

Section 4.—Change to read as follows:

4. The structural shapes shall conform to the chemical composition limits prescribed in Table I. Conformance shall be determined by the manufacturer by analyzing samples taken at the time the ingots are poured, or samples taken from the finished or semi-finished product. If the manufacturer has determined the chemical composition of the material during the course of manufacture, he shall not be required to sample and analyze the finished product.

Section 5.—Change the heading to "Sampling for Determination of Chemical Composition" and change as indicated above for Section 9 of Specification B 361, except exclude the note.

Section 6.—Change the heading to "Methods for Determination of Chemical Composition" and change as indicated above for Section 10 of Specification B 361.

Table I.—Change Footnote *a* to read as indicated above for Footnote *a* in Table I of Specification B 373.

Table II.—Change the tensile requirements for alloy 6351-T6 to the following:

Thickness, in.	Up through 0.124	0.125 – 0.749
Tensile strength, min, psi	42 000	42 000
Yield strength, min, psi	37 000	37 000
Elongation, min, per cent	8	10

Table III.—Change to read as shown in the accompanying Table IV.[3]

Section 8.—Change specimen dimensions as indicated above for Section 8 of Specification B 210.

Section 12.—Delete Paragraph (*d*).

Section 13.—Change as indicated above for Section 14 of Specification B 210.

Section 14.—Change as indicated above for Section 16 of Specification B 211.

Standard Specification for Aluminum-Alloy Round Welded Tubes (B 313 – 61):

Sections 4, 5, and 6.—Change as indicated above for the same sections in Specification B 210.

Table I.—Change Footnote *a* to read as indicated above for Footnote *a* in Table I of Specification B 373.

Section 15.—Change as indicated above for Section 14 of Specification B 210.

Section 16.—Change as indicated above for Section 16 of Specification B 211.

Standard Specification for Aluminum-Alloy Rivet and Cold Heating Wire and Rods (B 316 – 61):

Section 4.—Change to read as follows:

4. The rods and wire shall conform to the chemical composition limits prescribed in Table I. Conformance shall be determined by the manufacturer by analyzing samples taken at the time the ingots are poured, or samples taken from the finished or semi-finished product. If the manufacturer has determined the chemical composition of the material during the course of manufacture, he shall not be required to sample and analyze the finished product.

Section 5.—Change the heading to "Sampling for Determination of Chemical Composition" and change as indicated above for Section 9 of Specification B 361, except exclude the note.

Section 6.—Change the heading to "Methods for Determination of Chemical Composition" and change as indicated above for Section 10 of Specification B 361.

Table I.—Change Footnote *a* to read as indicated above for Footnote *a* in Table I of Specification B 373.

Section 8.—Change specimen dimensions in Paragraph (*a*) as indicated above for Section 8 of Specification B 210.

Section 15.—Change as indicated above for Section 16 of Specification B 210.

Standard Specification for Extruded Aluminum Alloy Bar, Rod, Pipe, and Structural Shapes for Electrical Purposes (Bus Conductors) (B 317 – 61):

Section 5.—Change to read as follows:

> 5. The bar, rod, pipe, and structural shapes shall conform to the chemical composition limits prescribed in Table I. Conformance shall be determined by the manufacturer by analyzing samples taken at the time the ingots are poured, or samples taken from the finished or semi-finished product. If the manufacturer has determined the chemical composition of the material during the course of manufacture, he shall not be required to sample and analyze the finished product.

Section 12.—Change the heading to "Sampling for Determination of Chemical Composition" and change as indicated above for Section 9 of Specification B 361, except exclude the note.

Section 13.—Change specimen dimensions in Paragraph (*a*) as indicated above for Section 8 of Specification B 210.

Section 14.—Change the heading of Paragraph (*a*) to "Chemical Composition" and change to read as indicated above for Section 10 of Specification B 361.

Standard Specification for Aluminum-Alloy Pipe for Gas and Oil Transmission and Distribution Piping Systems (B 345 – 61):

Section 4.—Change as indicated above for Section 4 of Specification B 241.

Section 5.—Change the heading to "Sampling for Determination of Chemical Composition" and change as indicated above for Section 9 of Specification B 361, except number the note as note 1.

Section 6.—Change the heading to "Methods for Determination of Chemical Composition" and change as indicated above for Section 10 of Specification B 361.

Table I.—Change Footnote *c* to read as indicated above for Footnote *a* in Table I of Specification B 373.

Table II.—Change the tensile requirements for alloy 6351-T6 as indicated above for Table II of Specification B 241, and change Footnote *b* to read as indicated above for Footnote *a* in Table I of Specification B 373.

Section 8.—Add the following to the end of this section:

> In no case shall the dimensions of the smallest specimen be less than the following:

Diameter of reduced section, in.	$\frac{1}{4}$
Length of reduced section, in.	$1\frac{1}{4}$
Radius of fillet, in.	$\frac{3}{16}$
Diameter of end section, in.	$\frac{3}{8}$
Over-all length, in.:	
With shouldered ends	$2\frac{3}{8}$
With threaded ends	3
With plain cylindrical ends	4

Section 17.—Change as indicated above for Section 14 of Specification B 210 except number the note as note 4.

Section 18.—Change as indicated above for Section 16 of Specification B 210.

Section 19.—Change as indicated above for Section 16 of Specification B 211.

Withdrawal of Standards

Standard Specification for Aluminum-Alloy Extruded Tubes (B 235 - 62):

The requirements of this Specification are recommended for incorporation in the revision of the Standard Specification for Aluminum-Alloy Extruded Bars, Rods, and Shapes (B 221), as covered earlier in this report.

Standard Specification for Type A and Type B Aluminum-Alloy Drawn Annealed Seamless Coiled Tubes (B 318 - 62):

The requirements of this specification are recommended for incorporation in the revision of the Standard Specification for Aluminum-Alloy Drawn Seamless Coiled Tubes for Special Purpose Applications (B 307), as covered earlier in this report.

American Standards

The committee recommends that revisions of the following 13 standard specifications, as covered earlier in this report, be accepted by the American Standards Assn. as American Standard: B 179, B 209, B 210, B 211, B 221, B 234, B 241, B 247, B 307, B 308, B 313, B 316, and B 345.

APPENDIX II

DESIGNATIONS FOR METALS AND ALLOYS ASSIGNED IN CONFORMANCE WITH RECOMMENDED PRACTICE FOR CODIFICATION OF LIGHT METALS AND ALLOYS (B 275)

COMPILED BY W. G. SNYDER, SECRETARY OF SUBCOMMITTEE VII ON CODIFICATION OF LIGHT ALLOYS

Designations for metals and alloys assigned in conformance with the Recommended Practice for Codification of Light Metals and Alloys, Cast and Wrought (B 275), and the ASTM Specifications in which they are used, are shown in Tables I, II, and III.

TABLE I.—ALUMINUM BASE.

Designation	ASTM Specification						
	B 26 – 62 T	B 37 – 60	B 85 – 60	B 108 – 62 T	B 179 – 62	B 285 – 61 T	B 327 – 60
850A	..	*	..	..	..	..	..
900A	..	*	..	..	..	..	..
920A	..	*	..	..	..	..	..
950A	..	*	..	..	..	..	..
980A	..	*	..	..	..	..	..
995A	..	..	..	..	*	..	..
C4A	*	..	..	..	*	*	..
CG100A	*	..	..	*	*	..	..
CG181A	..	..	..	..	..	..	*
CN42A	*	..	..	*	*	*	..
CS42A	[b]	..	..	*	*	..	..
CS43A	*	..	..	..	*	..	..
CS66A	..	..	..	*	*	..	..
CS72A	*	..	..	*	*	..	..
CS104A	..	..	..	*	*	..	..
G1C	..	..	..	..	..	..	*
G4A	*	..	..	..	*	..	..
G8A	..	..	*	..	*	..	..
G10A	*	..	..	..	*	..	..
GM70B	*	..	..	*	*	..	..
GS31A	..	..	..	..	*	..	..
GS42A	*	..	..	*	*	..	..
GZ42A	..	..	..	*	*	..	..

TABLE I.—ALUMINUM BASE (*Continued*).

Designation	ASTM Specification						
	B 26 – 62 T	B 37 – 60	B 85 – 60	B 108 – 62 T	B 179 – 62	B 285 – 61 T	B 327 – 60
S5A	*	..	..	*	*	..	..
S5B	*	..	..	*	*	[a]	..
S5C	..	..	*	..	*	..	..
S12A	..	..	*	..	*	..	..
S12B	..	..	*	..	*	..	..
S12C	..	..	..	..	*	..	..
SC51A	*	..	..	*	*	*	..
SC51B	..	..	..	*	*	..	..
SC51C	..	..	..	..	*	..	..
SC64A	..	..	..	[a]	[b]	..	..
SC64B	[a]	..	..	[a]	[b]	..	..
SC64C	[a]	..	..	[a]	*	..	..
SC64D	*	..	..	*	*	..	..
SC82A	*	..	..	..	*	..	..
SC84A	..	..	*	..	*	..	..
SC84B	..	..	*	..	*	..	..
SC84C	..	..	..	..	*	..	..
SC94A	..	..	..	[c]	[c]	..	..
SC103A	..	..	..	*	*	..	..
SC114A	..	..	*	..	*	..	..
SC122A	..	..	..	*	*	..	..
SF101A	..	..	..	..	*	..	..
SG70A	*	..	..	*	*	*	..
SG70B	..	..	..	*	*	..	..
SG70C	..	..	..	..	*	..	..
SG71A	..	..	..	..	*	..	..
SG100A	..	..	*	..	*	..	..
SG100B	..	..	*	..	*	..	..
SN122A	..	..	..	*	*	..	..
ZC60A	..	..	..	*	*	..	..
ZC81A	*	..	..	..	*	..	..
ZC81B	..	..	..	*	*	..	..
ZC32A	*	..	..	*	*	..	..
ZG42A	*	..	..	*	*	..	..
ZG61A	*	..	..	..	*	*	..
ZG61B	*	..	..	..	*	..	..
ZG71A	..	..	..	..	..	..	*

[a] Last appeared in 1957 issue.
[b] Last appeared in 1958 issue.
[c] Proposed for inclusion in 1963 issue.

TABLE II.—MAGNESIUM BASE.

Note.—Designations in italic were assigned for use by the producer, but are not in ASTM specifications.

Designation	ASTM Specification								
	B 80 – 62	B 90 – 61	B 91 – 60	B 92 – 61	B 93 – 59	B 94 – 58	B 107 – 61	B 199 – 62	B 217 – 60
9980A	..	..	..	*	..	..	..	..	..
9990A	..	..	..	*	..	..	..	..	..
9990B	..	..	..	*	..	..	..	..	..
9995A	..	..	..	*	..	..	..	..	..
A3A	..	..	..	..	..	..	..	..	..
AM80A	..	..	..	..	[a]	..	..	..	..
AM100A	*	..	..	..	*	..	..	*	..
AM100B	..	..	..	..	[a]	..	..	..	..
AZ10A	..	..	..	..	..	..	..	..	..
AZ31A	..	[a]	..	..	..	..	..	..	..
AZ31B	..	*	*	..	..	..	*	..	*
AZ31C	..	*	..	..	..	..	*	..	*
AZ61A	..	..	*	..	..	..	*	..	*
AZ63A	*	..	..	..	*	..	..	..	..
AZ80A	..	..	*	..	..	..	*	..	..
AZ81A	*	..	..	..	*	..	..	*	..
AZ91A	..	..	..	..	*	*	..	..	..
AZ91B	..	..	..	..	*	*	..	..	..
AZ91C	*	..	..	..	*	..	..	*	..
AZ92A	*	..	..	..	*	..	..	*	..
EK30A	[b]	..	..	..	..	..	..	..	..
EK41A	[b]	..	..	..	..	..	..	[b]	..
EZ33A	*	..	..	..	..	..	..	*	..
HK31A	*	*	..	..	..	..	..	*	..
HM21A	..	*	..	..	..	..	..	..	..
HM31A	..	..	..	..	..	..	..	..	..
HZ32A	*	..	..	..	..	..	..	..	..
K1A	*	..	..	..	..	..	..	..	..
M1A	..	..	..	..	..	..	*	..	*
M1B	..	..	..	..	[a]	..	..	..	..
TA54A	..	..	*	..	..	..	..	..	..
QE22A	*	..	..	..	..	..	..	*	..

TABLE II.—MAGNESIUM BASE (*Continued*).

Note.—Designations in italic were assigned for use by the producer, but are not in ASTM specifications.

Designation	ASTM Specification								
	B 80 – 62	B 90 – 61	B 91 – 60	B 92 – 61	B 93 – 59	B 94 – 58	B 107 – 61	B 199 – 62	B 217 – 60
ZE10A	..	*	..	..	..	..	..	..	..
ZE41A	*	..	..	..	..	..	..	..	..
ZH62A	*	..	..	..	..	..	..	..	..
ZK21A	..	..	..	..	..	..	..	..	..
ZK51A	*	..	..	..	..	..	..	..	..
ZK60A	..	..	*	..	..	..	*	..	*
ZK60B	..	..	..	..	..	..	..	..	..
ZK61A	*	..	..	..	..	..	..	..	..

[a] Last appeared in 1957 issue.
[b] Last appeared in 1961 issue.

TABLE III.—OTHERS.

Designation	ASTM Specification			
	B 176 – 62	B 102 – 52	B 86 – 57 T	B 240 – 57 T
Copper Base:				
Z30A	*	..	..	..
ZS144A	*	..	..	..
ZS331A	*	..	..	..
Lead Base:				
Y10A	..	*	..	..
YT155A	..	*	..	..
Tin Base:				
CY44A	..	*	..	..
PY1815A	..	*	..	..
YC135A	..	*	..	..
Zinc Base:				
AC41A	..	..	*	*
AG40A	..	..	*	*

REPORT OF COMMITTEE B-8

ON

ELECTRODEPOSITED METALLIC COATINGS AND RELATED FINISHES*

Committee B-8 on Electrodeposited Metallic Coatings and Related Finishes held two meetings during the year: on Sept. 27, 1962, in Washington, D. C., and Feb. 8, 1963, in Montreal, Canada.

The membership of the committee totals 141, of whom 52 are classified as producers, 16 as consumers, 73 as general interest members, and 12 as consulting members.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee B-8 requests approval by the Society at the Annual Meeting of the following recommendations. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] The existing specifications, definitions, and recommended practices referred to appear in their present form in the 1961 Book of ASTM Standards, Part 2, and the methods appear in the 1961 Book of ASTM Standards, Part 3, or in the 1962 Supplements thereto.

New Tentative

as appended hereto:[2]

Tentative Specification for Electrodeposited Coatings of Multi-Layer Nickel Plus Chromium on Zinc and Zinc-Base Alloys.

Revision of Tentatives

Tentative Definitions of Terms Relating to Electroplating (B 374 - 62 T):

Change the following definitions to read as indicated:

Dispersing Agent.—A material that increases the stability of a suspension of particles in a liquid medium.

Emulsion.—A suspension of fine particles or globules of one or more liquids in another liquid.

Ion Exchange.—A reversible process by which ions are interchanged between a solid and a liquid with no substantial structural changes of the solid.

Mandrel.—A form used as a cathode in electroforming; a mold or matrix.

Matrix.—See *Mandrel.*

Plastisol.—A suspension of a finely divided resin in a plasticizer, that can be converted to a continuous film by the application of heat. Distinct from baking enamels, etc., in that substantially all the original mixture becomes a part of the film; there is no significant evaporation of solvent. The films are usually much thicker than obtainable from coatings which depend on the evaporation of a volatile solvent.

Sequestering Agent.—An agent that forms soluble complex compounds with, or sequesters, a simple ion, thereby suppressing the activity of that ion. Thus, in water treatment the effects of hardness can be suppressed by adding agents to sequester calcium and magnesium. See *Chelating Agent.*

Surface Active Agent.—A substance that affects markedly the interfacial or surface tension of solutions even when present in very low concentrations.

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[2] The new tentative appears in the 1963 Supplement to *Book of ASTM Standards*, Part 2.

Tentative Specifications for Electrodeposited Coatings of Multi-Layer Nickel plus Chromium on Steel (B 375 – 61 T):

Section 5(b).—Change the first sentence to begin: "Two recommended corrosion tests are the Method . . . ."

Revision of Standard and Reversion to Tentative

Standard Method of Test for Sealing of Anodically Coated Aluminum (**B 136 – 45**):

Revise and revert to tentative as appended hereto.[3]

Tentatives Continued Without Revision

Tentative Specifications for:

Chromate Finishes on Electrodeposited Zinc, Hot-Dipped Galvanized, and Zinc Die-Cast Surfaces (**B 201 – 55 T**), and

Electrodeposited Coatings of Nickel and Chromium on Steel (**A 166 – 61 T**).

Tentative Recommended Practices for:

Cleaning Metals Prior to Electroplating (**B 322 – 58 T**), and

Preparation of Nickel for Electroplating with Nickel (**B 343 – 60 T**).

Editorial Change

Standard Method of Test for Dielectric Strength of Anodically Coated Aluminum (**B 110 – 45**):

Change Note 2 to read as follows:

NOTE 2.—A power transformer with a secondary voltage adequate to cause breakdown of the anodic coating will be required (such voltages may be of the order of 5000 V). Safety precautions for working with high voltages sources should be observed.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Papers, Specifications, and Definitions (F. Ogburn, chairman) prepared the revised Definitions B 374 referred to earlier in the report.

Section G (R. B. Saltonstall, chairman) prepared the new Tentative Specifications for Electrodeposited Coatings of Multi-Layer Nickel Plus Chromium on Zinc and Zinc-Base Alloys appended to this report.

Subcommittee II on Performance Tests (W. H. Safranek, chairman).—The 1963 report of Section A on Program No. 4 on Performance of Copper, Nickel, and Chromium Coatings has been prepared.[4] This report supplements the Program No. 4 report published in the 1961 *Proceedings*.

Section E on Performance of Tin Coatings is working on solderability tests of tin on copper.

Subcommittee III on Conformance Tests (L. C. Borchert, chairman).—Papers are being prepared by section chairmen for the symposium to be given at the 1963 Convention of the American Electroplaters' Society.

Subcommittee IV on Electroplating Practice (R. A. Ehrhardt, chairman) drafted a new Tentative Recommended Practice for Treating Aluminum Mandrels Used in Electroforming.

Subcommittee VI on Anodic Oxidation (W. C. Cochran, chairman) prepared the revision of Method B 136 as appended to this report. J. Andrus is chairman of a group planning a Symposium on Anodizing of Aluminum for the Spring Committee Week meeting in 1964.

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[3] The revised method appears in the 1963 Supplement to *Book of ASTM Standards*, Part 3.

[4] See p. 215.

At the Montreal meeting, the committee voted unanimously to form a new Subcommittee VII on Electroforming. Former Section E of Subcommittee IV forms the basis of this new subcommittee with A. D. Squitero, chairman.

The committee voted favorably on a motion to participate in the activities of the International Organization for Standardization's new Technical Committee 107 (ISO/TC 107) through the American Standards Assn. (ASA).

This report has been submitted to letter ballot of the committee, which consists of 141 voting members; 98 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

C. H. SAMPLE,
Chairman.

R. E. HARR,
General Secretary.

APPENDIX

PERFORMANCE OF DECORATIVE NICKEL-CHROMIUM COATINGS ON STEEL

REPORT OF SECTION A, SUBCOMMITTEE II ON PERFORMANCE TESTS

A continuing study of the performance of decorative nickel-chromium coatings has been made under Program No. 4 by Subcommittee II. The principal objectives of this program were to determine: (1) effect of nickel and copper strikes under bright nickel, (2) effect of type and thickness of chromium, and (3) effect of buffing bright nickel. Comparisons of buffed Watts nickel versus bright nickel, and of copper-nickel - chromium versus nickel - chromium were also made. Some 26 lots of panels were plated for this program; they were exposed on April 28, 1956, at Kure Beach and on May 1, 1956, at Detroit.

Corrodkote and CASS test[1] ratings, inspection data for 4 and 5 yr, Magne-Gage thickness measurements on individual panels, and a graphic presentation of outdoor exposure are detailed in Tables I to V. The following notations apply:

Types of Failure

pR—Pinpoint rusting.
cR—Crater rusting.
Rs—Rust stain.
Z—Crazing.
Sp—Surface pitting.
E—Spangled or etched in appearance (non-uniform cracking).
B—Blistering.
W—Crows feet (multi-branched cracks).
S—Stains or spots other than that of obvious basis metal corrosion products.
F—Flaking.
vs—Very slight in amount.
s—Slight in amount.
i—Intermediate in amount.
x—Excessive in amount.
vx—Very excessive in amount.
e—Edge condition.
f—Faint.

Conclusions supplementing those in Appendix I to the 1961 report of Committee B-8[2] are also presented.

Discussion:

The 5-yr exposure data show good agreement for panels plated with nickel by platers X and Y when the chromium was plated by plater W. Lots 9 through 19 are representative of this good agreement. Very good agreement was obtained from lots 12 and 15 chromium plated by plater W. On the other hand, lots 1 and 23, chromium-plated by platers X and Y, which also were plated with 0.000010 in. of chromium, showed inconsistent results.

Conclusions:

1. The exposure data for 4 and 5 yrs support the conclusions in Appendix I to the 1961 report of Committee B-8, which included exposure data through three years.
2. Unintended variations in the

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[1] Method of Copper-Accelerated Acetic Acid-Salt Spray (Fog) Testing (Cass Test) (B 368), Method of Corrosion Testing of Decorative Chromium Plating by the Corrodkote Procedure (B 380), *1961 Book of ASTM Standards*, Part 3.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 61, p. 334 (1961).

TABLE I.—CASS AND CORRODKOTE TEST RATINGS FOR PROGRAM NO. 4 PANELS.

Panel	CASS	Corrodkote	Panel	CASS	Corrodkote	Panel	CASS	Corrodkote
Lot No. 1 Plater X			Lot No. 4 Plater Y			Lot No. 8 Plater X		
No. 25	3/3	3/3	No. 22	3/2	9/6	No. 22	3/3	2/2
No. 36	8/8	6/6	No. 52	2/2	9/7	No. 13	6/6	3/3
No. 34	8/8	8/8	No. 32	4/4	10/7	No. 21	5/5	3/3
No. 14	5/5	6/6	No. 23	3/3	9/7	No. 35	8/8	3/3
No. 35	5/5	4/4	No. 11	5/5	9/7	No. 11	3/3	4/4
Ave.	6/6	5/5	Ave.	3/3	9/7	Ave.	5/5	3/3
Lot No. 1 Plater Y			Lot No. 5 Plater X			Lot No. 8 Plater Y		
No. 41	3/3	4/4	No. 14	5/4	5/4	No. 21	3/3	8/7
No. 12	4/4	8/8	No. 23	3/3	8/6	No. 33	4/4	10/9
No. 13	8/7	9/7	No. 25	2/2	5/3	No. 52	3/2	9/8
No. 23	5/5	8/6	No. 32	2/2	10/8	No. 23	4/3	9/8
No. 32	6/6	9/7	No. 36	2/2	5/3	No. 13	4/3	8/7
Ave.	5/5	8/6	Ave.	3/3	7/5	Ave.	4/3	9/8
Lot No. 2 Plater X			Lot No. 5 Plater Y			Lot No. 9 Plater X		
No. 33	3/3	4/3	No. 11	4/2	10/7	No. 24	10/9	10/8
No. 23	5/4	5/4	No. 43	9/7	10/9	No. 11	9/9	10/10
No. 26	2/2	3/3	No. 32	9/7	10/8	No. 12	9/8	9/8
No. 34	3/3	3/3	No. 33	9/7	9/7	No. 16	10/9	10/9
No. 12	3/3	4/3	No. 21	3/3	9/7	No. 25	9/8	10/9
Ave.	3/3	4/3	Ave.	7/5	10/8	Ave.	9/9	10/9
Lot No. 2 Plater Y			Lot No. 6 Plater X			Lot No. 9 Plater Y		
No. 53	9/8	10/9	No. 22	6/6	2/2	No. 42	9/9	9/6
No. 12	8/7	10/8	No. 33	7/7	3/3	No. 21	9/8	9/7
No. 11	9/8	10/9	No. 13	5/5	2/2	No. 63	9/7	10/8
No. 21	3/3	4/4	No. 23	4/4	2/2	No. 32	8/7	9/7
No. 33	3/3	9/8	No. 11	3/3	3/3	No. 12	8/6	9/7
Ave.	6/6	9/8	Ave.	5/5	2/2	Ave.	9/7	9/7
Lot No. 3 Plater X			Lot No. 6 Plater Y			Lot No. 10 Plater X		
No. 13	3/3	2/2	No. 53	8/6	9/7	No. 31	10/8	10/8
No. 15	6/5	3/3	No. 32	8/7	10/8	No. 15	10/8	10/8
No. 12	4/4	2/2	No. 43	4/3	5/4	No. 33	10/8	10/8
No. 36	8/7	4/3	No. 42	3/3	5/3	No. 13	10/10	10/10
No. 26	4/4	2/2	No. 52	8/6	8/6	No. 14	9/7	10/8
Ave.	5/5	3/2	Ave.	6/5	7/6	Ave.	10/8	10/8
Lot No. 3 Plater Y			Lot No. 7 Plater X			Lot No. 10 Plater Y		
No. 21	8/7	8/7	No. 24	6/5	6/5	No. 32	8/7	9/7
No. 12	6/4	9/7	No. 33	5/5	4/4	No. 41	9/7	9/7
No. 32	9/7	9/7	No. 11	9/9	4/4	No. 22	9/7	10/8
No. 31	5/3	9/7	No. 35	8/8	6/6	No. 53	9/7	10/8
No. 33	8/6	10/8	No. 22	5/5	4/4	No. 11	8/7	9/7
Ave.	7/5	9/7	Ave.	7/6	5/5	Ave.	9/7	9/7
Lot No. 4 Plater X			Lot No. 7 Plater Y			Lot No. 11 Plater X		
No. 12	3/2	10/8	No. 51	4/4	5/5	No. 32	3/3	9/9
No. 34	3/3	9/7	No. 22	3/3	9/7	No. 14	4/3	3/2
No. 15	3/3	10/7	No. 32	7/6	10/8	No. 12	1/1	3/3
No. 22	4/4	10/7	No. 33	3/2	3/2	No. 26	2/2	3/2
No. 33	5/4	9/6	No. 41	2/2	4/3	No. 23	1/1	2/2
Ave.	4/3	10/7	Ave.	4/3	6/5	Ave.	2/2	4/4

TABLE I.—*Continued.*

Panel	CASS	Corrodkote	Panel	CASS	Corrodkote	Panel	CASS	Corrodkote
Lot No. 11 Plater Y			Lot No. 15 Plater X			Lot No. 17 Plater Y		
No. 13	7/7	6/6	No. 36	4/4	6/6	No. 21	9/7	5/4
No. 52	4/4	3/3	No. 11	3/3	3/3	No. 12	9/8	8/7
No. 51	3/3	3/3	No. 23	2/2	5/5	No. 52	8/6	7/5
No. 33	2/2	9/7	No. 14	2/2	6/5	No. 43	6/5	9/7
No. 12	3/2	9/7	No. 25	5/5	10/9	No. 41	8/7	9/7
Ave.	4/4	6/5	Ave.	3/3	6/6	Ave.	8/7	8/6
Lot No. 12 Plater X			Lot No. 15 Plater Y			Lot No. 17 Plater Z		
No. 34	3/3	3/3	No. 13	3/3	3/3	No. 7	9/8	5/4
No. 12	2/2	3/3	No. 42	5/4	5/4	No. 1	9/7	9/7
No. 15	4/4	3/3	No. 51	4/4	6/4	No. 6	10/8	6/4
No. 32	1/1	2/2	No. 22	5/5	5/4	No. 2	9/7	10/8
No. 21	3/3	3/3	No. 32	3/3	4/3	No. 14	10/8	10/8
Ave.	3/3	3/3	Ave.	4/4	5/4	Ave.	9/8	8/6
Lot No. 12 Plater Y			Lot No. 15 Plater Z			Lot No. 18 Plater X		
No. 32	9/7	3/3	No. 10	9/9	7/7	No. 22	9/8	8/7
No. 33	5/4	3/3	No. 1	9/8	7/6	No. 26	6/4	4/3
No. 43	2/1	3/1	No. 11	9/8	6/5	No. 21	3/3	4/4
No. 21	5/4	6/4	No. 12	9/8	9/8	No. 11	7/6	9/8
No. 42	4/3	3/2	No. 15	9/8	6/5	No. 35	9/7	9/7
Ave.	5/4	4/3	Ave.	9/8	7/6	Ave.	7/6	7/6
Lot No. 13 Plater X			Lot No. 16 Plater X			Lot No. 18 Plater Y		
No. 35	9/8	9/7	No. 34	9/8	8/7	No. 3A	9/8	4/2
No. 16	9/8	9/8	No. 36	9/8	9/8	No. 5A	8/7	6/4
No. 26	9/7	9/7	No. 35	8/7	4/3	No. 5B	9/7	6/4
No. 15	9/8	10/8	No. 21	9/7	5/3	No. 1A	7/5	6/4
No. 13	9/8	10/9	No. 16	9/8	5/4	No. 1B	9/7	6/4
Ave.	9/8	9/8	Ave.	9/8	6/5	Ave.	8/7	6/4
Lot No. 13 Plater Y			Lot No. 16 Plater Y			Lot No. 19 Plater X		
No. 42	9/7	10/7	No. 23	8/6	7/5	No. 34	7/7	10/7
No. 53	9/7	10/8	No. 52	8/7	8/6	No. 31	4/4	4/4
No. 51	9/7	9/7	No. 12	5/4	8/6	No. 22	4/4	4/4
No. 13	9/8	9/7	No. 53	6/5	4/3	No. 12	9/8	4/4
No. 22	7/5	9/7	No. 41	8/7	9/7	No. 25	3/3	4/4
Ave.	9/7	9/7	Ave.	7/6	7/5	Ave.	5/5	5/5
Lot No. 14 Plater X			Lot No. 16 Plater Z			Lot No. 19 Plater Y		
No. 35	9/8	10/8	No. 7	9/8	9/7	Panels not available.		
No. 41	9/7	9/7	No. 4	10/9	9/7			
No. 36	9/8	9/7	No. 14	9/8	10/9			
No. 14	9/7	10/8	No. 12	9/8	9/7			
No. 22	9/7	10/8	No. 8	9/8	10/8			
Ave.	9/7	10/8	Ave.	9/8	9/8			
Lot No. 14 Plater Y			Lot No. 17 Plater X			Lot No. 20B Plater X		
No. 52	9/7	10/7	No. 26	8/6	7/5	No. 11	7/7	10/9
No. 12	8/6	10/8	No. 11	7/5	6/4	No. 32	9/8	6/5
No. 11	8/6	9/7	No. 36	9/7	8/6	No. 24	9/9	10/9
No. 13	9/8	10/7	No. 23	5/3	8/6	No. 23	9/9	9/9
Ave.	9/7	10/7	No. 16	9/7	10/8	No. 36	9/7	9/7
			Ave.	8/6	8/6	Ave.	9/8	9/8

Continued on page 218.

TABLE I.—*Concluded.*

Panel	CASS	Corrodkote	Panel	CASS	Corrodkote	Panel	CASS	Corrodkote
Lot No. 20	Plater Y		Lot No. 22	Plater X		Lot No. 23	Plater Y	
No. 13.......	9/9	10/8	No. 32.......	10/10	5/5	No. 11.......	4/3	5/3
No. 12.......	9/9	10/10	No. 15.......	10/9	6/5	No. 33.......	4/3	9/7
No. 11.......	9/9	9/7	No. 22.......	10/10	7/5	No. 52.......	3/2	6/4
No. 52.......	9/9	9/9	No. 12.......	9/8	6/4	No. 23.......	4/3	5/3
No. 22.......	5/4	10/10	No. 13.......	8/7	8/5	No. 22.......	4/3	9/7
Ave........	8/8	10/9	Ave........	9/9	6/6	Ave........	4/3	7/5
Lot No. 21	Plater X		Lot No. 22	Plater Y				
No. 22.......	5/5	5/4	No. 43.......	9/7	10/8			
No. 11.......	3/3	3/3	No. 12.......	9/7	10/9			
No. 32.......	5/5	4/4	No. 51.......	9/7	9/7			
No. 31.......	5/5	5/5	No. 13.......	9/7	10/8			
No. 14.......	4/4	5/5	No. 52.......	8/7	9/7			
Ave........	4/4	4/4	Ave........	9/7	10/8			
Lot No. 21	Plater Y		Lot No. 23	Plater X				
No. 41.......	1/1	2/2	No. 14.......	4/4	3/3			
No. 21.......	2/2	2/2	No. 26.......	3/3	5/5			
No. 13.......	1/1	2/2	No. 12.......	3/3	3/3			
No. 31.......	3/3	5/4	No. 34.......	3/3	3/3			
No. 22.......	1/1	2/2	No. 23.......	3/3	3/3			
Ave........	2/2	3/2	Ave........	3/3	3/3			

chromium plating techniques appear to have had more influence on the consistency of outdoor corrosion performance than the copper or nickel plating techniques.

3. The results of 16-hr CASS and Corrodkote tests revealed two trends recognized from the 3-yr exposure data. These trends were as follows:

(*a*) Chromium coatings having a thickness of 30 or 60 microinches were more protective than those having a thickness of 10 microinches, in the case of panels plated with either bright or buffed Watts-type nickel.

(*b*) Buffing bright nickel plate had a beneficial influence on the performance of a composite of 1.5 mils of bright nickel and 10 microinches of chromium. In this respect, CASS and Corrodkote supported Kure Beach exposure data. Such buffing appeared to have no effect on panels exposed at Detroit.

4. CASS and Corrodkote data showed good agreement for most of the panels prepared by platers *X* and *Y*. Differences in CASS and Corrodkote results for *X* and *Y* panels that included copper were less significant than the differences observed between *X* and *Y* panels exposed at Detroit and Kure Beach.

Other Programs:

Program No. 5, Steel Panels, was exposed in November, 1960 to: (1) evaluate the effects of increases in the thickness of the chromium over double layered nickel, (2) compare different types of chromium, and (3) compare double layered nickel with bright nickel.

Program No. 6, Aluminum Panels, was exposed in October, 1961, to study the influence of variations in the thicknesses of copper and double layered nickel.

Program No. 7, Zinc-Alloy Die-Cast Panels, was exposed in November, 1961, to investigate the effects of variations in the thicknesses of copper, semibright nickel (in a double layered system) and chromium.

Respectfully submitted,

VINCENT J. CASSIDY, *Chairman*
Section A, Subcommittee II.

TABLES II-V.—on pp. 220–239.

TABLE II.—INSPECTION DATA PROGRAM NO. 4, FOUR AND FIVE YEARS EXPOSURE.

Lot	Plate and Thickness, mils	Plater	Panel	Detroit, Mich.						Panel	Kure Beach, N. C.					
				Four Year (5/1/56 to 5/12/60)			Five Year (5/1/56 to 5/12/61)				Four Year (4/29/56 to 5/9/60)			Five Year (4/28/56 to 5/15/61)		
				Ratings		Description	Ratings		Description		Ratings		Description	Ratings		Description
				Protection	Appearance		Protection	Appearance			Protection	Appearance		Protection	Appearance	
No. 1....	1.5 Bright nickel 0.01 Regular chromium	X	No. 24	5	5	ipR, sRs, eZ	6	6	ipR, sRs	No. 23	0	0	xpR, xRs, icR	0	0	xpR, xRs, icR
			No. 21	5	5	ipR, sRs, eZ	6	6	ipR, sRs	No. 32	0	0	xpR, xRs, icR	0	0	xpR, xRs, icR
			No. 13	5	5	ipR, sRs, eZ	6	6	ipR, sRs	No. 15	0	0	xpR, xRs, icR	0	0	xpR, xRs, icR
			No. 31	5	5	ipR, sRs, eZ	6	6	ipR, sRs	No. 16	0	0	xpR, sRs, icR	0	0	xpR, sRs, icR
			No. 26	5	5	ipR, sRs, eZ	5	5	xpR, sRs							
		Y	No. 4-2	4	4	xpR, sRs, eZ	5	4	xpR, sRs	No. 1-1	0	0	xpR, xRs	0	0	xpR, xRs
			No. 5-3	4	4	xpR, sRs, eZ	5	4	xpR, sRs	No. 2-1	0	0	xpR, xRs	0	0	xpR, xRs
			No. 3-3	4	4	xpR, sRs, eZ	5	4	xpR, sRs	No. 5-2	0	0	xpR, xRs	0	0	xpR, xRs
			No. 5-1	4	4	xpR, sRs, eZ	5	4	xpR, sRs	No. 4-3	0	0	xpR, xRs	0	0	xpR, xRs
			No. 2-2	4	4	xpR, sRs, eZ	5	4	xpR, sRs							
No. 2....	0.05 Copper 1.45 Bright nickel 0.01 Chromium	X	No. 16	4	4	xpR, iB	3	3	xpR, xB	No. 15	0	0	Rated 0 at 2 years	0	0	Rated 0 at 2 years
			No. 11	4	4	xpR, iB	3	3	xpR, xB							
			No. 31	5	5	xpR, sB	3	3	xpR, xB	No. 35	0	0	Rated 0 at 2 years	0	0	Rated 0 at 2 years
			No. 13	4	4	xpR, xB	3	3	xpR, xB							
			No. 22	4	4	xpR, iB	3	3	xpR, xB	No. 32	0	0	Rated 0 at 2 years	0	0	Rated 0 at 2 years
										No. 25	0	0	Rated 0 at 2 years	0	0	Rated 0 at 2 years
		Y	No. 4-2	4	3	xpR, iSp, xRs	3	0	xpR, xRs, xB, xS	No. 3-1	9	8	vspR, sSp	9	6	spR, xSp, vsB
										No. 3-2	9	8	vspR, sSp	9	4	spR, xSp
			No. 4-3	4	3	xpR, iSp, xRs, sB	3	0	xpR, xRs, xB, xS	No. 2-3	9	8	vspR, sSp	9	7	spR, xSp
			No. 5-1	4	3	xpR, iSp, xRs, iB	3	0	xpR, xRs, xB, xS							
			No. 4-1	4	3	xpR, iSp, xRs, xB	3	0	xpR, xRs, xB, xS							
			No. 5-2	4	3	xpR, iSp, xRs	3	0	xpR, xRs, xB, xS							

No. 3....	0.05 Low pH nickel 1.45 Bright nickel 0.01 Regular chromium	X	No. 33	4	3	xpR, iSp, sRs	6	5	xpR, sRs	No. 14	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 34	4	3	xpR, iSp, sRs	6	5	xpR, sRs							
			No. 22	4	3	xpR, iSp, sRs	6	5	xpR, sRs	No. 16	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 32	4	3	xpR, iSp, sRs	6	5	xpR, sRs							
			No. 31	4	3	xpR, iSp, sRs	6	5	xpR, sRs	No. 21	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
										No. 24	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
		Y	No. 2-3	4	3	xpR, iSp, sRs	4	3	xpR, iRs	No. 1-	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 5-1	3	2	xpR, iSp, sRs	4	3	xpR, iRs							
			No. 4-2	4	3	xpR, iSp, sRs	4	3	xpR, iRs	No. 1-1	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 4-3	4	3	xpR, iSp, sRs	4	3	spR, iRs							
			No. 2-2	4	3	xpR, iSp, sRs	4	1	xpR, xS	No. 5-2	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
										No. 1-3	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
No. 4....	0.15 Copper 0.65 Acid copper 0.7 Bright nickel 0.01 Regular chromium	X	No. 24	2	2	sRs, icR, xB (broken)	1	0	xpR, scR	No. 11	0	0	vxcR	0	0	vxcR
										No. 14	0	0	vxcR	0	0	vxcR
			No. 26	2	2	sRs, icR, xB (broken)	1	0	xpR, scR	No. 25	0	0	vxcR	0	0	vxcR
										No. 35	0	0	vxcR	0	0	vxcR
			No. 23	2	2	sRs, icR, xB (broken)	1	0	xpR, scR							
			No. 31	2	2	sRs, icR, xB (broken)	1	0	xpR, scR							
			No. 16	2	2	sRs, icR, xB (broken)	1	0	xpR, scR							
		Y	No. 4-3	2	2	iRs, xSp, xcR, xB	6	3	vscR, spR, xRs, xB, xSp	No. 3-1	8	6	spR, iSp	7	3	ipR, xSp
										No. 1-3	10	8	iSp	10	5	xSp
			No. 4-1	2	2	sRs, xSp, xcR, xB	1	0	vscR, xpR, xRs, xB, xSp	No. 5-3	9	7	spR, iSp	9	5	spR, xSp
			No. 4-2	2	2	sRs, xSp, xcR, xB	1	0	vscR, xpR, xRs, xB, xSp							
			No. 3-3	2	2	sRs, xSp, xcR, xB	1	0	vscR, xpR, xRs, xB, xSp							
			No. 2-1	2	2	sRs, xSp, xcR, xB	1	0	vscR, xpR, xRs, xB, xSp							

TABLE II.—*Continued.*

Lot	Plate and Thickness, mils	Plater	Panel	Detroit, Mich.						Panel	Kure Beach, N. C.					
				Four Year (5/1/56 to 5/12/60)			Five Year (5/1/56 to 5/12/61)				Four Year (4/29/56 to 5/9/60)			Five Year (4/28/56 to 5/15/61)		
				Ratings		Description	Ratings		Description		Ratings		Description	Ratings		Description
				Protection	Appearance		Protection	Appearance			Protection	Appearance		Protection	Appearance	
No. 5....	0.15 Nickel (pH 2.5) 0.65 Acid copper 0.7 Bright nickel 0.01 Regular chromium	X	No. 21	6	5	xpR, sRs	3	2	xpR, sRs	No. 11	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 26	6	5	xpR, sRs	3	2	xpR, sRs	No. 15	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 13	6	5	xpR, sRs	3	2	xpR, sRs	No. 22	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 12	6	5	xpR, sRs	3	2	xpR, sRs	No. 31	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 35	5	4	xpR, sRs	3	2	xpR, sRs							
		Y	No. 2-3	4	4	xpR, sSp	5	2	xpR, xRs, iSp	No. 3-1	0	0	xpR, xcR, xRs, sSp	0	0	xpR, xcR, xRs, sSp
			No. 2-2	4	4	xpR, sSp, scR	5	2	xpR, xRs, iSp	No. 5-1	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 1-2	4	4	xpR, sSp, scR	5	2	xpR, xRs, iSp	No. 1-3	8	6	spR, iSp	8	7	spR, sSp
			No. 4-2	4	4	xpR, sSp	5	2	xpR, xRs, iSp							
			No. 5-2	4	4	xpR, sSp	5	2	xpR, xRs, iSp							
No. 6....	0.15 Nickel (pH 2.5) 1.35 Bright nickel 0.01 Regular chromium	X	No. 36	2	2	xpR, xRs	5	5	xpR	No. 15	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 34	2	2	xpR, xRs	5	5	xpR	No. 16	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 25	2	2	xpR, xRs	5	5	xpR	No. 24	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 21	2	2	xpR, xRs	5	5	xpR	No. 32	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 14	2	2	xpR, xRs	5	5	xpR							
		Y	No. 2-1	4	3	xpR, xRs	4	3	xpR, sRs, sB	No. 3-1	3	1	vxpR, xRs	1	0	vxpR, xRs
			No. 2-2	4	3	xpR, xRs	4	3	xpR, sRs	No. 4-1	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 3-3	4	3	xpR, xRs	4	3	xpR, sRs, sB	No. 1-2	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 5-1	4	3	xpR, xRs	3	1	xpR, sRs, xS	No. 2-3	0	0	vxpR, xRs	0	0	vxpR, xRs
			No. 1-1	4	3	xpR, xRs	4	3	xpR, sRs							

No. 7....	0.15 Copper 1.35 Bright nickel 0.01 Regular chromium	X	No. 23	3	2	xRs, iSp, xB (broken)	2	1	iRs, sSp, xB	No. 12	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 26	3	2	xRs, iSp, xB (broken)	2	1	iRs, sSp, xB	No. 25	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 32	3	2	xRs, iSp, xB (broken)	2	1	iRs, sSp, xB	No. 34	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 13	3	2	xRs, iSp, xB (broken)	2	1	iRs, sSp, xB	No. 16	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 36	3	2	xRs, iSp, xB (broken)	2	1	iRs, sSp, xB							
		Y	No. 3-1	4	3	xpR, xRs, xSp	4	2	xpR, xRs, xSp, iB	No. 1-1	6	4	vspR, sSp, xeF	6	3	vspR, xSp, xeF
										No. 2-1	1	0	xpR, xRs, icR, xSp	1	0	xpR, xRs, icR, xSp
			No. 5-2	3	2	xpR, xRs, xSp	4	0	xpR, xRs, xSp, iB, xS	No. 4-2	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 2-3	4	3	xpR, xRs, xSp	4	2	xpR, xRs, xSp, iB							
			No. 4-3	4	3	xpR, xRs, xSp	4	2	xpR, xRs, xSp, iB							
			No. 1-3	4	3	xpR, xRs, xSp	4	2	xpR, xRs, xSp, iB							
No. 8....	1.5 Bright nickel 0.005 Regular chromium	X	No. 14	2	2	xpR, xRs	6	6	ipR, sRs	No. 24	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 23	2	2	xpR, xRs	6	6	ipR, sRs							
			No. 34	2	2	xpR, xRs	6	6	ipR, sRs	No. 25	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 12	2	2	xpR, xRs	6	6	ipR, sRs							
			No. 15	2	2	xpR, xRs	6	6	ipR, sRs	No. 36	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
										No. 33	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
		Y	No. 1-1	2	2	xpR, xRs	4	3	xpR, sRs	No. 4-1	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 5-3	2	2	xpR, xRs	4	3	xpR, sRs							
			No. 3-1	2	2	xpR, xRs	4	3	xpR, sRs	No. 5-1	2	1	xpR, xRs	0	0	xpR, xRs
			No. 4-2	2	2	xpR, xRs	4	3	xpR, sRs	No. 1-2	3	1	xpR, xRs	2	0	xpR, xRs
			No. 2-2	2	2	xpR, xRs	4	3	xpR, sRs	No. 4-3	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
No. 9....	1.5 Bright nickel 0.03 Regular chromium	X	No. 21	10	9	Z	9	8	vspR, xZ	No. 13	8	6	spR, Z	5	2	xpR, xZ
			No. 15	10	9	Z	9	8	vspR, xZ	No. 22	5	3	ipR, Z	3	0	xpR, xZ
			No. 32	10	9	Z	10	8	xZ	No. 26	3	1	xpR, Z	2	1	xpR, xZ
			No. 33	10	9	Z	10	8	xZ	No. 31	2	1	xpR, Z	2	1	xpR, xZ
			No. 35	10	9	Z	10	8	xZ							

TABLE II.—*Continued.*

Lot	Plate and Thickness, mils	Plater	Panel	Detroit, Mich.						Panel	Kure Beach, N. C.					
				Four Year (5/1/56 to 5/12/60)			Five Year (5/1/56 to 5/12/61)				Four Year (4/29/56 to 5/9/60)			Five Year (4/28/56 to 5/15/61)		
				Ratings		Description	Ratings		Description		Ratings		Description	Ratings		Description
				Protection	Appearance		Protection	Appearance			Protection	Appearance		Protection	Appearance	
No. 9— *Continued*		Y	No. 5-1	10	9	Z	9	7	vspR, xZ, W	No. 4-1	7	5	spR, Z	6	3	xpR, xZ
			No. 1-3	10	9	Z	10	7	xZ	No. 5-3	5	3	ipR, Z	4	2	xpR, xZ
			No. 1-1	10	9	Z	9	7	vspR, xZ, W	No. 2-3	7	5	spR, Z	6	3	xpR, xZ
			No. 3-3	10	9	Z	10	7	xZ	No. 4-3	5	3	ipR, Z	4	2	xpR, xZ
			No. 2-2	10	9	Z	10	7	xZ							
No. 10....	1.5 Bright nickel 0.06 Regular chromium	X	No. 24	9	7	vspR, E	10	6	E, xZ	No. 12	8	5	spR, E	7	4	spR, xE
			No. 34	9	7	vspR, E	10	6	E, xZ	No. 21	7	4	spR, E	7	4	spR, xE
			No. 35	9	7	vspR, E	10	6	E, xZ	No. 23	7	4	spR, E	7	4	spR, xE
			No. 25	9	7	vspR, E	10	6	E, xZ	No. 26	8	5	spR, E	8	5	spR, xE
			No. 11	9	7	vspR, E	10	6	E, xZ							
		Y	No. 1-2	9	8	vspR, Z	10	8	xZ	No. 2-1	7	5	spR, E	5	2	xpR, xZ
			No. 5-1	9	8	vspR, Z	10	8	xZ	No. 3-1	7	5	spR, Z	5	2	xpR, xZ
			No. 1-3	9	8	vspR, Z	10	8	xZ	No. 4-2	5	3	ipR, Z	5	2	xpR, xZ
			No. 5-2	9	8	vspR, Z	10	8	xZ	No. 2-3	7	5	spR, Z	6	3	ipR, xZ
			No. 4-3	9	8	vspR, Z	10	8	xZ							
No. 11....	1.5 Bright nickel 0.005 CR110	X	No. 35	2	2	xpR, xRs	5	4	xpR, sRs	No. 11	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 31	2	2	xpR, xRs	5	4	xpR, sRs							
			No. 25	2	2	xpR, xRs	5	4	xpR, sRs	No. 13	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 22	2	2	xpR, xRs	5	4	xpR, sRs							
			No. 16	2	2	xpR, xRs	5	4	xpR, sRs	No. 24	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years

		Y	No. 1-1	2	2	xpR, xRs	4	3	xpR, sRs	No. 4-1	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 3-1	2	2	xpR, xRs	4	3	xpR, sRs	No. 2-2	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 4-3	2	2	xpR, xRs	4	3	xpR, sRs	No. 2-3	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 3-2	2	2	xpR, xRs	4	3	xpR, sRs	No. 5-3	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 2-1	2	2	xpR, xRs	4	3	xpR, sRs							
No. 12....	1.5 Bright nickel 0.01 CR110	X	No. 13	3	3	xpR, xRs	6	6	xpR	No. 14	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 35	3	3	xpR, xRs	6	6	xpR	No. 16	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 31	3	3	xpR, xRs	6	6	xpR	No. 25	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 36	3	3	xpR, xRs	6	6	xpR	No. 24	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 26	3	3	xpR, xRs	6	6	xpR							
		Y	No. 1-1	3	3	xpR, xRs	6	5	xpR, sSp	No. 4-1	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 1-3	3	3	xpR, xRs	6	5	xpR, sSp	No. 5-1	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 3-1	3	3	xpR, xRs	6	5	xpR, sSp	No. 2-2	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 5-3	3	3	xpR, xRs	6	5	xpR, sSp	No. 2-3	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 5-2	3	3	xpR, xRs	6	5	xpR, sSp							
No. 13....	1.5 Bright nickel 0.03 CR110	X	No. 31	9	8	vspR, Z	9	8	vspR, xZ	No. 12	6	4	ipR, Z	3	1	xpR, sRs, xZ
			No. 21	9	8	vspR, Z	9	8	vspR, xZ	No. 24	6	4	ipR, Z	4	2	xpR, sRs, xZ
			No. 11	9	8	vspR, Z	9	8	vspR, xZ	No. 23	5	3	ipR, Z	3	1	xpR, sRs, xZ
			No. 36	9	8	vspR, Z	9	8	vspR, xZ	No. 32	6	4	ipR, Z	3	2	xpR, sRs, xZ
			No. 22	9	8	vspR, Z	9	8	vspR, xZ							
		Y	No. 3-1	9	8	vspR, Z	9	8	vspR, xZ	No. 1-1	7	5	spR, Z	7	4	ipR, xZ
			No. 4-3	9	8	vspR, Z	9	8	vspR, xZ	No. 2-1	7	5	spR, Z	3	1	ipR, xcR, xZ
			No. 1-2	9	8	vspR, Z	9	8	vspR, xZ	No. 3-2	6	4	ipR, Z	6	3	ipR, xZ
			No. 4-1	9	8	vspR, Z	9	8	vspR, xZ	No. 5-2	6	4	ipR, Z	6	3	ipR, xZ
			No. 2-3	9	8	vspR, Z	9	8	vspR, xZ							

TABLE II.—*Continued.*

Lot	Plate and Thickness, mils	Plater	Panel	Detroit, Mich.						Panel	Kure Beach, N. C.					
				Four Year (5/1/56 to 5/12/60)			Five Year (5/1/56 to 5/12/61)				Four Year (4/29/56 to 5/9/60)			Five Year (4/28/56 to 5/15/61)		
				Ratings		Description	Ratings		Description		Ratings		Description	Ratings		Description
				Protection	Appearance		Protection	Appearance			Protection	Appearance		Protection	Appearance	
No. 14....	1.5 Bright nickel 0.06 CR110	X	No. 33	9	8	vspR, Z	9	7	vspR, xZ	No. 11	6	4	ipR, E	4	1	xpR, iRs, xE
			No. 26	9	8	vspR, Z	9	7	vspR, xZ	No. 21	5	3	ipR, E	4	1	xpR, iRs, xE
			No. 15	9	8	vspR, Z	9	7	vspR, xZ	No. 23	7	5	ipR, E	5	2	xpR, iRs, xE
			No. 24	9	8	vspR, Z	9	7	vspR, xZ	No. 34	4	2	xpR, E	4	1	xpR, iRs, xE
			No. 31	9	8	vspR, Z	9	7	vspR, xZ							
		Y	No. 2-3	9	7	vspR, E	9	7	vspR, E, xZ	No. 2-1	7	5	ipR, E	7	4	ipR, xRs, xE
			No. 3-3	9	7	vspR, E	9	7	vspR, E, xZ	No. 3-2	7	5	ipR, E	6	3	ipR, xRs, xE
			No. 4-3	9	7	vspR, E	9	7	vspR, E, xZ	No. 4-2	6	4	ipR, E	5	2	ipR, xRs, xE
			No. 3-1	9	7	vspR, E	9	7	vspR, E, xZ	No. 5-3	6	4	ipR, E	5	2	ipR, xRs, xE
			No. 5-1	9	7	vspR, E	9	7	xspR, E, xZ							
No. 15....	1.5 Watts nickel buffed 0.01 Regular chromium	X	No. 34	7	5	spR, xSp, sRs	8	4	spR, sRs, xSp	No. 26	0	0	xpR, xRs, xSp	0	0	xpR, xRs, xSp
			No. 12	7	5	spR, xSp, sRs	8	4	spR, sRs, xSp	No. 24	0	0	xpR, xRs, xSp	0	0	xpR, xRs, xSp
			No. 16	6	4	spR, xSp, sRs	8	4	spR, sRs, xSp	No. 31	0	0	xpR, xRs, xSp	0	0	xpR, xRs, xSp
			No. 35	7	5	spR, xSp, sRs	8	4	spR, sRs, xSp	No. 32	0	0	xpR, xRs, xSp	0	0	xpR, xRs, xSp
			No. 21	7	5	spR, xSp, sRs	8	4	spR, sRs, xSp							
		Y	No. 2-3	7	4	ipR, xSp, sRs	8	4	sRs, spR, xSp	No. 1-2	2	1	xpR, xRs, iSp	0	0	xpR, xRs, iSp
			No. 1-1	8	5	ipR, xSp, sRs	8	4	sRs, spR, xSp	No. 3-3	0	0	xpR, xRs, iSp	0	0	xpR, xRs, iSp
			No. 2-1	8	5	ipR, xSp, sRs	8	4	sRs, spR, xSp	No. 4-3	0	0	xpR, xRs, iSp	0	0	xpR, xRs, iSp
			No. 4-1	6	3	ipR, xSp, sRs	8	4	sRs, spR, xSp	No. 5-3	1	0	xpR, xRs, iSp	0	0	xpR, xRs, iSp
			No. 5-2	7	4	ipR, xSp, sRs	8	4	sRs, spR, xSp							
		Z	No. 7	5	3	ipR, xSp, iRs	8	4	sRs, spR, xSp	No. 6	0	0	xpR, xRs, scR	0	0	xpR, xRs, scR, iSp
			No. 13	8	4	spR, xSp, iRs	8	4	sRs, spR, xSp							
			No. 4	5	3	ipR, xSp, iRs	8	4	sRs, spR, xSp	No. 8	0	0	xpR, xRs, icR	0	0	xpR, xRs, icR, iSp
			No. 2	5	3	ipR, xSp, iRs	8	4	sRs, spR, xSp							
			No. 3	5	3	ipR, xSp, iRs	8	4	sRs, spR, xSp	No. 9	1	0	xpR, xRs	0	0	xpR, xRs, icR, iSp
										No. 14	5	4	ipR, iRs, scR	4	2	xpR, iRs, scR, iSp

No. 16....	1.5 Watts nickel buffed 0.03 Regular chromium	X	No. 23	9	8	vspR, vsRs, Z	9	8	vspR, xZ	No. 12	0	0	xpR, xRs, Z	0	0	xpR, xRs, Z
			No. 33	9	8	vspR, vsRs, Z	9	8	vspR, xZ	No. 13	0	0	xpR, xRs, Z	0	0	xpR, xRs, Z
			No. 31	9	8	vspR, vsRs, Z	9	8	vspR, xZ	No. 24	0	0	xpR, xRs, Z	0	0	xpR, xRs, Z
			No. 26	9	8	vspR, vsRs, Z	9	8	vspR, xZ	No. 25	0	0	xpR, xRs, Z	0	0	xpR, xRs, Z
			No. 14	9	8	vspR, vsRs, Z	9	8	vspR, xZ							
		Y	No. 4-3	9	7	vspR, vsRs, Z	9	7	vspR, vsRs, xZ	No. 2-1	5	4	ipR, Z	5	3	xpR, Z
			No. 4-2	9	7	vspR, vsRs, Z	9	7	vspR, vsRs, xZ	No. 2-2	5	4	ipR, Z	5	3	xpR, Z
			No. 3-2	9	7	vspR, vsRs, Z	9	7	vspR, vsRs, xZ	No. -3	7	5	ipR, Z	6	4	xpR, Z
			No. 5-1	9	7	vspR, vsRs, Z	9	7	vspR, vsRs, xZ	No. 3-3	6	4	ipR, Z	6	4	xpR, Z
			No. 3-1	8	6	vspR, vsRs, Z	9	7	vspR. vsRs, xZ							
		Z	No. 13	9	7	vspR. vsRs, Z	10	7	xZ	No. 5	8	7	spR, Z	8	6	spR, iZ
			No. 15	9	7	vspR, vsRs, Z	10	7	xZ	No. 9	8	7	spR, Z	8	6	spR, iZ
			No. 6	9	7	vspR, vsRs, Z	10	7	xZ	No. 10	8	7	spR, Z	8	6	spR, iZ
			No. 3	9	7	vspR, vsRs, Z	10	7	xZ	No. 11	9	8	vspR, Z	9	7	vspR, iZ
			No. 2	9	7	vspR, vsRs, Z	10	7	xZ							
No. 17....	1.5 Watts nickel buffed 0.06 Regular chromium	X	No. 15	8	5	ipR, sRs, E	8	5	vsRs, spR, E, xZ	No. 14	0	0	xpR, xRs, E	0	0	xpR, xRs, E
			No. 33	8	5	ipR, sRs, E	8	5	vsRs, spR, E, xZ	No. 24	0	0	xpR, xRs, E	0	0	xpR, xRs, E
			No. 21	8	5	ipR, sRs, E	8	5	vsRs, spR, E, xZ	No. 31	0	0	xpR, xRs, E	0	0	xpR, xRs, E
			No. 34	8	5	ipR, sRs, E	8	5	vsRs, spR, E, xZ	No. 32	0	0	xpR, xRs, E	0	0	xpR, xRs, E
			No. 25	8	5	ipR, sRs, E	8	5	vsRs, spR, E, xZ							
		Y	No. 5-3	9	7	ipR, sRs, E	9	7	vspR, E, xZ	No. 3-1	5	4	ipR, iRs, Z	4	2	xpR, iRs, xZ
			No. 5-1	9	7	ipR, sRs, E	9	7	vspR, E, xZ	No. 2-2	7	6	ipR, iRs, Z	7	5	ipR, iRs, xZ
			No. 3-3	9	7	ipR, sRs, E	9	7	vspR, E, xZ	No. 1-3	7	6	ipR, iRs, Z	6	4	xpR, iRs, xZ
			No. 3-2	9	7	ipR, sRs, E	9	7	vspR, E, xZ	No. 2-3	6	5	ipR, iRs, Z	6	4	xpR, iRs, xZ
			No. 4-2	9	7	ipR, sRs, E	9	7	vspR, E, xZ							
		Z	No. 11	9	6	ipR, sRs, E	9	6	vspR, xE	No. 4	2	0	xpR, xRs, E	3	1	xpR, xRs, xE
			No. 5	9	6	ipR, sRs, E	9	6	vspR, xE	No. 8	6	4	ipR, iRs, E	6	4	ipR, iRs, xE
			No. 13	9	6	ipR, sRs, E	9	6	vspR, xE	No. 15	8	6	ipR, iRs, E	8	6	spR, iRs, xE
			No. 9	9	6	ipR, sRs, E	9	6	vspR, xE							
			No. 12	9	6	ipR, sRs, E	9	6	vspR, xE							
No. 18....	1.5 Bright nickel 0.01 Crack-free chromium, buffed	X	No. 33	2	0	xpR, xRs	5	3	xpR, iRs, xSp	No. 12	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 32	2	0	xpR. xRs	5	3	xpR, iRs, xSp							
			No. 13	2	0	xpR, xRs	5	3	xpR, iRs, xSp	No. 24	1	0	xpR, xRs	0	0	xpR, xRs
			No. 23	2	0	xpR, xRs	5	3	xpR, iRs, xSp	No. 31	0	0	xpR, xRs	0	0	xpR, xRs
			No. 15	2	0	xpR, xRs	5	3	xpR, iRs, xSp							

TABLE II.—*Continued.*

Lot	Plate and Thickness, mils	Plater	Panel	Detroit, Mich.						Panel	Kure Beach, N. C.					
				Four Year (5/1/56 to 5/12/60)			Five Year (5/1/56 to 5/12/61)				Four Year (4/29/56 to 5/9/60)			Five Year (4/28/56 to 5/15/61)		
				Ratings		Description	Ratings		Description		Ratings		Description	Ratings		Description
				Protection	Appearance		Protection	Appearance			Protection	Appearance		Protection	Appearance	
No. 18—*Continued*		Y	No. 2A	4	4	xpR, xRs	5	4	xpR, iRs	No. 4A	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 2B	4	4	xpR, xRs	5	4	xpR, iRs							
			No. 4C	4	4	xpR, xRs	5	4	xpR, iRs	No. 4B	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 3C	4	4	xpR, xRs	5	4	xpR, iRs							
			No. 2C	4	4	xpR, xRs	5	4	xpR, iRs	No. 5C	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
No. 19....	1.5 Bright nickel 0.06 Crack-free chromium, buffed	X	No. 14	6	5	ipR, sSp	7	6	ipR, sRs	No. 16	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 13	6	5	ipR, sSp	7	6	ipR, sRs							
			No. 35	6	5	ipR, sSp	7	6	ipR, sRs	No. 26	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 36	6	5	ipR, sSp	7	6	ipR, sRs							
			No. 24	6	5	ipR, sSp	7	6	ipR, sRs	No. 33	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
		Y	No. 4A	7	6	ipR, sSp	8	7	spR, sRs	No. 1A	1	0	xpR, xRs	2	1	xpR, xRs
			No. 3B	7	6	ipR, sSp	8	7	spR, sRs	No. 5A	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 5B	7	6	ipR, sSp	8	7	spR, sRs							
			No. 2A	7	6	ipR, sSp	8	7	spR, sRs	No. 1C	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
No. 20B...	1.5 Bright nickel buffed 0.01 Regular chromium	X	No. 26	5	3	xpR, xRs, xSp	8	4	spR, xSp	No. 13	9	8	vspR, sSp	9	8	vspR, sSp
			No. 22	5	3	xpR, xRs, xSp	8	4	spR, xSp	No. 16	9	8	vspR, sSp	8	7	vspR, sSp
			No. 34	5	3	xpR, xRs, xSp	8	4	spR, xSp	No. 21	8	7	spR, sSp	7	6	spR, sSp
			No. 31	5	3	xpR, xRs, xSp	8	4	spR, xSp	No. 33	8	7	spR, sSp	8	7	spR, sSp
			No. 14	5	3	xpR, xRs, xSp	8	4	spR, xSp							

No. 20....		Y	No. 5-3	5	3	xpR, xRs, xSp	7	5	ipR, sRs, iSp	No. 2-1	4	2	ipR, iRs, xSp	3	0	xpR, iRs, xSp
			No. 3-2	5	3	xpR, xRs, xSp	7	5	ipR, sRs, iSp	No. 3-3	5	3	ipR, iRs, xSp	4	1	xpR, iRs, xSp
			No. 4-1	5	3	xpR, xRs, xSp	7	5	ipR, sRs, iSp	No. 4-3	5	3	ipR, iRs, xSp	3	0	xpR, iRs, xSp
			No. 3-1	5	3	xpR, xRs, xSp	7	5	ipR, sRs, iSp							
			No. 2-3	5	3	xpR, xRs, xSp	7	5	ipR, sRs, iSp							
No. 21....	0.75 Bright nickel 0.01 Regular chromium	X	No. 34	2	1	xpR, xRs	5	4	sRs, xpR	No. 23	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 12	2	1	xpR, xRs	5	4	sRs, xpR	No. 25	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 15	2	1	xpR, xRs	5	4	sRs, xpR	No. 33	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 24	2	1	xpR, xRs	5	4	sRs, xpR	No. 35	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 21	2	1	xpR, xRs	5	4	sRs, xpR							
		Y	No. 1-2	1	1	xpR, xRs	4	2	xpR, xRs	No. 3-2	0	0	vxpR, xRs	0	0	vxpR, xRs
			No. 3-3	1	1	xpR, xRs	4	2	xpR, xRs	No. 4-2	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 5-3	1	1	xpR, xRs	4	2	xpR, xRs	No. 2-3	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 5-2	1	1	xpR, xRs	4	2	xpR, xRs							
			No. 5-1	1	1	xpR, xRs	4	2	xpR, xRs							
No. 22....	2.5 Bright nickel 0.01 Regular chromium	X	No. 35	6	5	ipR, sSp, eZ	6	6	ipR, W	No. 21	0	0	vxpR, xRs	0	0	vxpR, xRs
			No. 33	6	5	ipR, sSp, eZ	6	6	ipR, W	No. 25	0	0	vxpR, xRs	0	0	vxpR, xRs
			No. 24	6	5	ipR, sSp, eZ	6	6	ipR, W	No. 34	0	0	vxpR, xRs	0	0	vxpR, xRs
			No. 26	6	5	ipR, sSp, eZ	6	6	ipR, W	No. 36	0	0	vxpR, xRs	0	0	vxpR, xRs
			No. 16	6	5	ipR, sSp, eZ	6	6	ipR, W							
		Y	No. 3-3	4	4	ipR, sSp, eZ	4	3	xpR, sRs	No. 1-1	7	5	spR, sRs, iSp, W	6	3	ipR, sRs, iSp, W
			No. 4-2	4	4	ipR, sSp, eZ	4	3	xpR, sRs	No. 2-1	4	2	ipR, iRs, iSp, W	4	1	ipR, iRs, iSp, W
			No. 2-2	4	4	ipR, sSp, eZ	4	3	xpR, sRs	No. 3-2	4	2	ipR, iRs, iSp, W	5	2	ipR, iRs, iSp, W
			No. 4-1	4	4	ipR, sSp, eZ	4	3	xpR, sRs	No. 5-3	1	0	xpR, xRs, W	1	0	xpR, xRs, W
			No. 3-1	4	4	ipR, sSp, eZ	4	3	xpR, sRs							
No. 23....	1.5 Bright nickel 0.01 Regular chromium	X	No. 36	2	2	xpR, xRs	6	6	ipR	No. 15	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 16	2	2	xpR, xRs	6	6	ipR	No. 25	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 24	2	2	xpR, xRs	6	6	ipR	No. 31	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 21	2	2	xpR, xRs	6	6	ipR	No. 33	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
			No. 35	2	2	xpR, xRs	6	6	ipR							

TABLE II.—*Concluded.*

Lot	Plate and Thickness, mils	Plater	Panel	Detroit, Mich.						Panel	Kure Beach, N. C.					
				Four Year (5/1/56 to 5/12/60)			Five Year (5/1/56 to 5/12/61)				Four Year (4/29/56 to 5/9/60)			Five Year (4/28/56 to 5/15/61)		
				Ratings			Ratings				Ratings			Ratings		
				Protection	Appearance	Description	Protection	Appearance	Description		Protection	Appearance	Description	Protection	Appearance	Description
No. 23—*Continued*		Y	No. 1-2	3	3	xpR, xRs	5	5	xpR	No. 3-1	0	0	vxpR, xRs	0	0	vxpR, xRs
			No. 4-1	3	3	xpR, xRs	5	5	xpR	No. 4-3	0	0	vxpR, xRs	0	0	vxpR, xRs
			No. 5-3	3	3	xpR, xRs	5	5	xpR	No. 5-1	0	0	vxpR, xRs	0	0	vxpR, xRs
			No. 3-2	3	3	xpR, xRs	5	5	xpR							
			No. 2-1	3	3	xpR, xRs	5	5	xpR							
No. 24[a] . . .	1.5 Watts nickel buffed Heavy crack-free chromium buffed	Z	. . .	. . .	. . .	. . .	. . .	. . .	. . .	AMIT	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
										AM2	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
										AM3	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
										AM5	0	0	Rated 0 at 3 years	0	0	Rated 0 at 3 years
No. 25[a] . . .	1.5 Watts nickel buffed Thin crack-free chromium buffed	Z	. . .	. . .	. . .	. . .	. . .	. . .	. . .	AM6	4	3	ipR, sRs, scR, sB, sSp, xeR, seB	6	5	ipR, sRs, scR, sB, xeR, sSp, seB
										AM7	7	6	ipR, sSp	7	6	ipR, sSp
										AM9	2	1	ipR, iB, iRs, iSp	2	1	ipR, iRs, iB, iSp
										AM10	8	7	spR, sSp, icpR	8	7	spR, iepR, sSp
No. 26[a] . . .	1.5 Watts nickel buffed Thin regular chromium	Z	. . .	. . .	. . .	. . .	. . .	. . .	. . .	AM11	5	4	ipR, sRs, iSp	4	2	xpR, iRs, iSp
										AM13	5	4	ipR, sRs, iSp	3	1	xpR, iRs, iSp
										AM14	6	5	ipR, sRs, iSp	4	2	xpR, iRs, iSp
										AM15	4	3	xpR, sRs, iSp	3	1	xpR, iRs, iSp

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[a] Lots 24, 25, and 26 used special steel.

TABLE III.—PROGRAM NO. 4, PERFORMANCE OF PANELS BY PLATERS X AND Y.

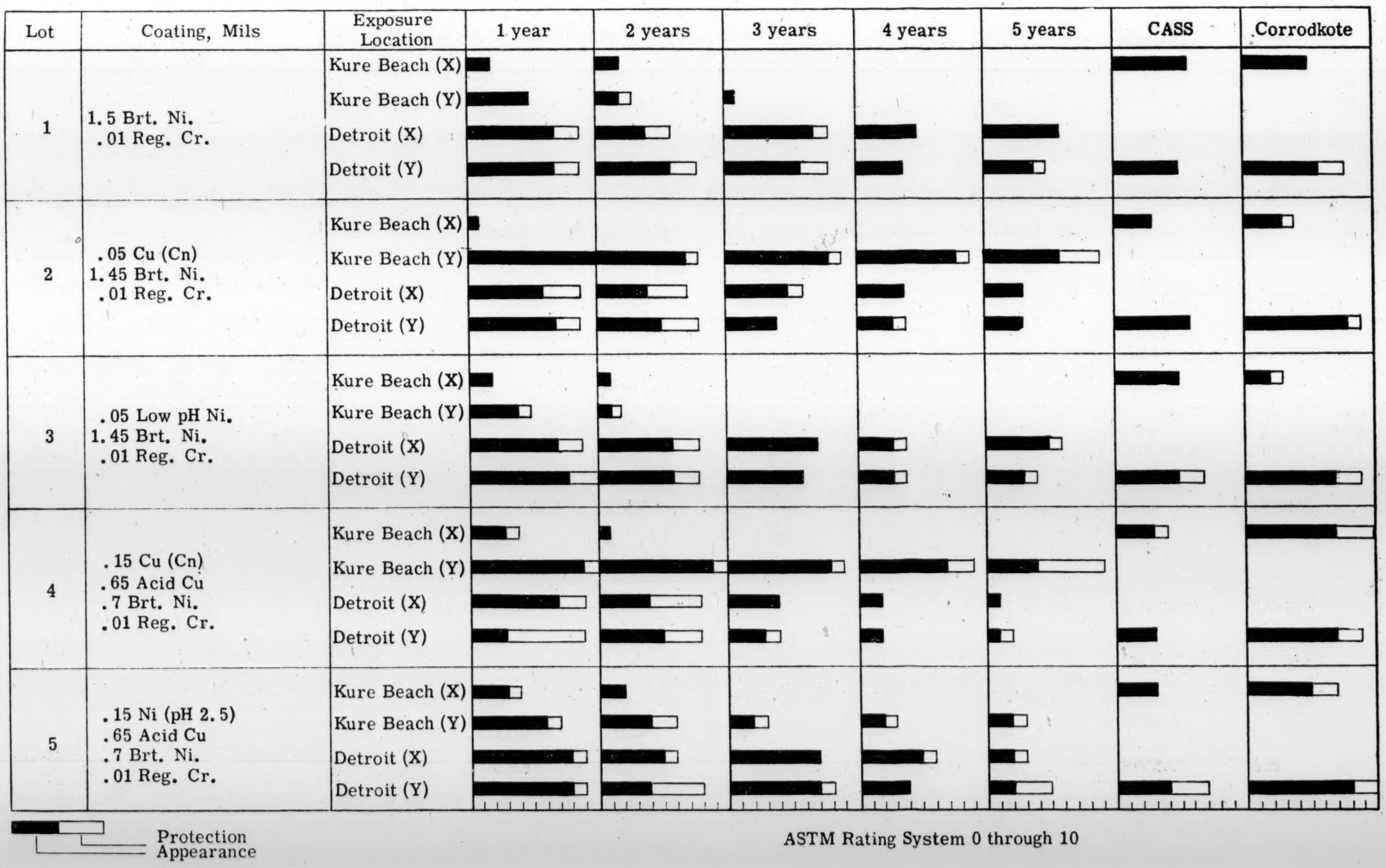
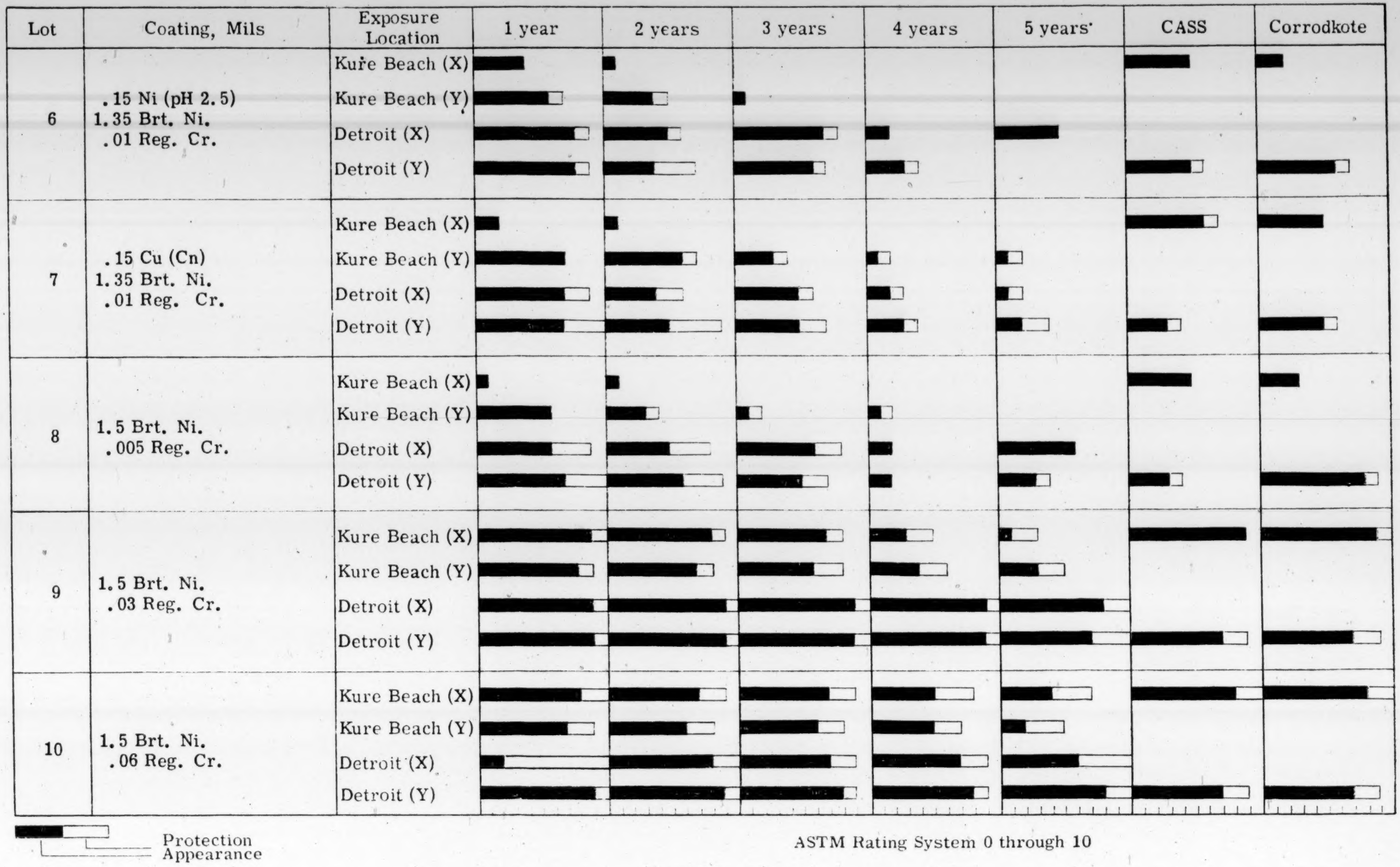


TABLE III.—*Continued.*



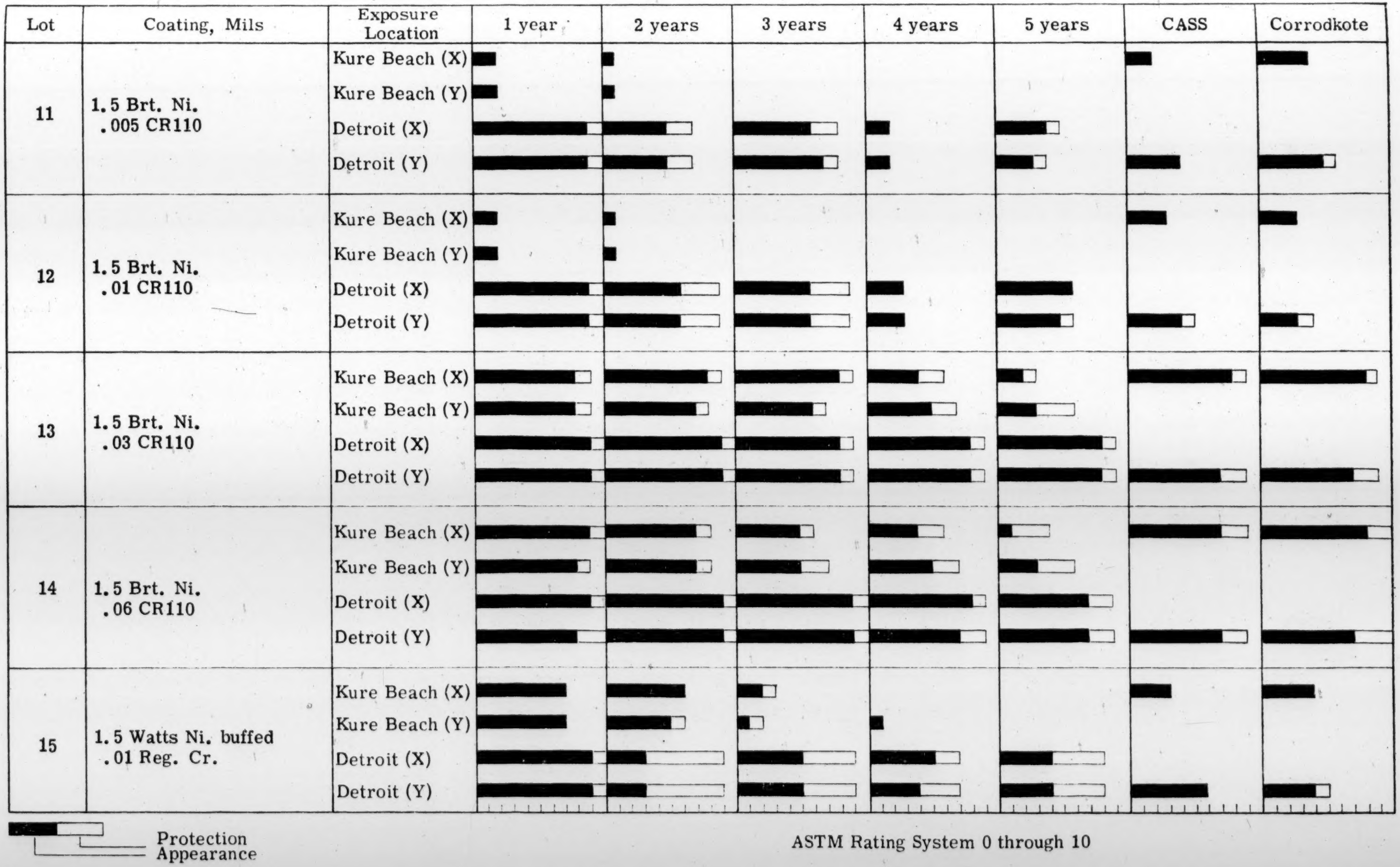
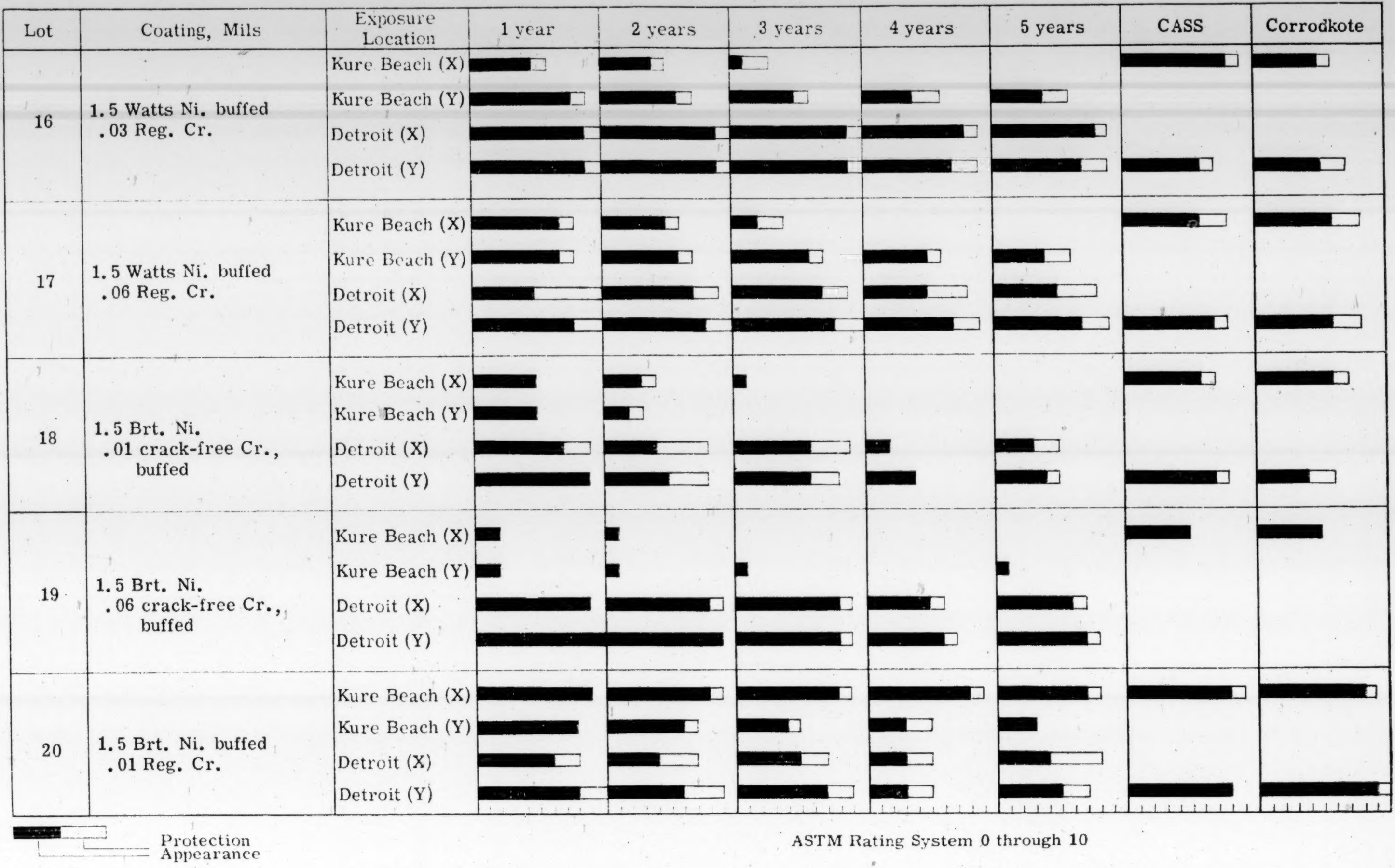

Lot
Coating, Mils
Exposure Location
1 year
2 years
3 years
4 years
5 years
CASS
Corrodkote
11
1. 5 Brt. Ni. .005 CR110
12
1. 5 Brt. Ni. .01 CR110
13
1. 5 Brt. Ni. .03 CR110
14
1. 5 Brt. Ni. .06 CR110
15
1. 5 Watts Ni. buffed .01 Reg. Cr.
Kure Beach (X)
Kure Beach (Y)
Detroit (X)
Detroit (Y)
Protection
Appearance
ASTM Rating System 0 through 10

TABLE III.—*Continued*



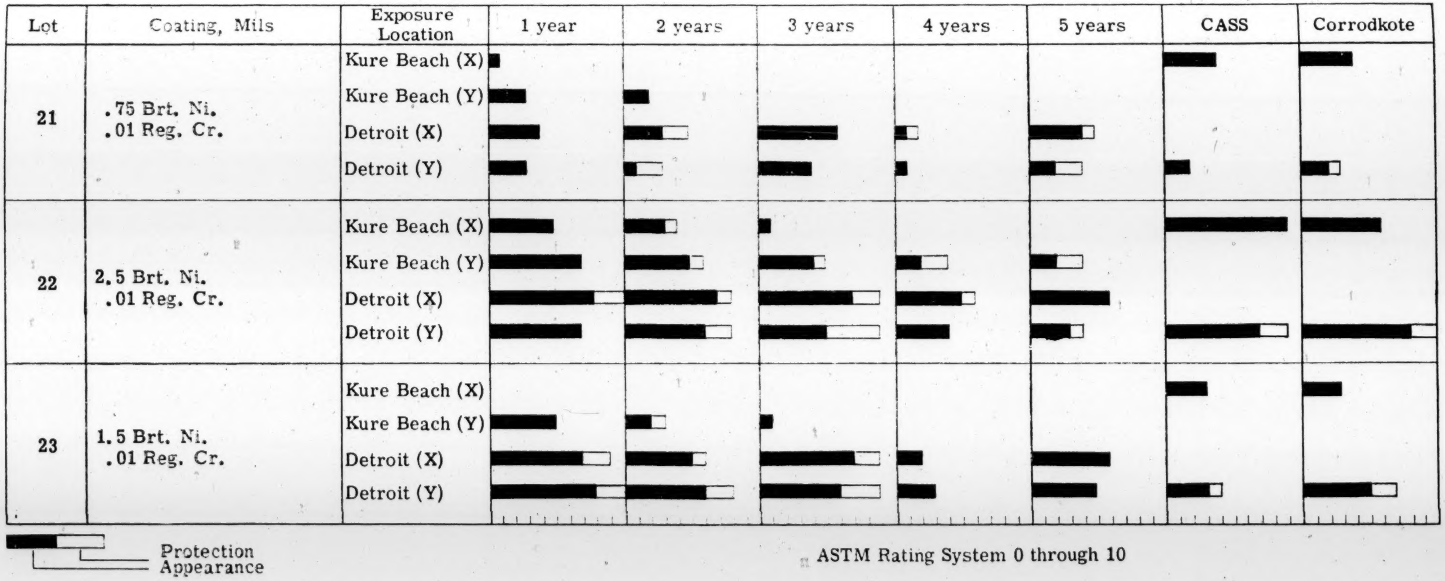

Lot
Coating, Mils
Exposure Location
1 year
2 years
3 years
4 years
5 years
CASS
Corrodkote
21
.75 Brt. Ni.
.01 Reg. Cr.
22
2.5 Brt. Ni.
.01 Reg. Cr.
23
1.5 Brt. Ni.
.01 Reg. Cr.
Kure Beach (X)
Kure Beach (Y)
Detroit (X)
Detroit (Y)
Protection
Appearance
ASTM Rating System 0 through 10

TABLE IV.—PLATING THICKNESS—MAGNE-GAGE PANELS EXPOSED IN DETROIT, MICH.

Panel	Nickel Thickness, mils	Panel	Nickel Thickness, mils	Panel	Nickel Thickness, mils
No. 1-26	1.57	No. 5-35	...	No. 10-11	1.35
No. 1-31	1.44	No. 5-12	...	No. 10-25	1.40
No. 1-13	1.36	No. 5-13	...	No. 10-35	1.41
No. 1-21	1.36	No. 5-26	...	No. 10-34	1.42
No. 1-24	1.41	No. 5-21	...	No. 10-24	1.49
No. 22-1	2.27	No. 52-5	...	No. 43-10	1.96
No. 51-1	1.43	No. 42-5	...	No. 52-10	1.93
No. 33-1	1.96	No. 22-5	...	No. 13-10	2.29
No. 42-1	1.77	No. 12-5	...	No. 51-10	2.20
No. 53-1	1.74	No. 23-5	...	No. 12-10	1.80
No. 1A-11	1.42	No. 6-14	1.43	No. 11-16	1.54
No. 1A-12	1.38	No. 6-21	1.38	No. 11-22	1.35
No. 1A-36	1.41	No. 6-25	1.48	No. 11-25	1.43
No. 1A-22	1.37	No. 6-34	1.43	No. 11-31	1.37
No. 1A-16	1.40	No. 6-36	1.42	No. 11-35	1.41
No. 1B-15	1.42	No. 11-6	1.88	No. 21-11	2.06
No. 1B-35	1.57	No. 51-6	1.50	No. 32-11	1.97
No. 1B-25	1.50	No. 33-6	2.13	No. 43-11	1.72
No. 1B-22	1.43	No. 22-6	2.20	No. 31-11	1.84
No. 1B-31	1.51	No. 21-6	1.90	No. 11-11	1.90
No. 2-22	1.48[a]	No. 7-36	1.79[a]	No. 12-26	1.34
No. 2-13	1.38[a]	No. 7-13	1.62[a]	No. 12-36	1.52
No. 2-31	1.45[a]	No. 7-32	1.60[a]	No. 12-31	1.48
No. 2-11	1.43[a]	No. 7-26	1.70[a]	No. 12-35	1.52
No. 2-16	1.45[a]	No. 7-23	1.62[a]	No. 12-13	1.49
No. 52-2	1.79[a]	No. 13-7	1.85[a]	No. 52-12	1.57
No. 41-2	1.50[a]	No. 43-7	1.50[a]	No. 53-12	1.82
No. 51-2	1.82[a]	No. 23-7	1.85[a]	No. 31-12	1.70
No. 43-2	1.53[a]	No. 52-7	1.45[a]	No. 13-12	1.96
No. 42-2	1.74[a]	No. 31-7	1.85[a]	No. 11-12	2.06
No. 3-31	1.40	No. 8-15	1.53	No. 13-22	1.33
No. 3-32	1.37	No. 8-12	1.58	No. 13-36	1.37
No. 3-22	1.33	No. 8-34	1.61	No. 13-11	1.48
No. 3-34	1.43	No. 8-23	1.48	No. 13-21	1.44
No. 3-33	1.40	No. 8-14	1.58	No. 13-31	1.39
No. 22-3	2.27	No. 22-8	2.15	No. 23-13	1.85
No. 43-3	1.62	No. 42-8	1.72	No. 41-13	1.51
No. 42-3	1.58	No. 31-8	1.91	No. 12-13	2.02
No. 51-3	1.57	No. 53-8	1.66	No. 43-13	1.72
No. 23-3	2.17	No. 11-8	2.02	No. 31-13	1.57
No. 4-16	...	No. 9-35	1.38	No. 14-31	1.40
No. 4-31	...	No. 9-33	1.38	No. 14-24	1.44
No. 4-28	...	No. 9-32	1.50	No. 14-15	1.39
No. 4-26	...	No. 9-15	1.46	No. 14-26	1.41
No. 4-24	...	No. 9-21	1.37	No. 14-33	1.42
No. 21-4	...	No. 22-9	1.93	No. 51-14	1.69
No. 33-4	...	No. 33-9	2.15	No. 31-14	2.33
No. 42-4	...	No. 11-9	2.02	No. 43-14	1.70
No. 41-4	...	No. 13-9	2.09	No. 33-14	2.37
No. 43-4	...	No. 51-9	2.00	No. 23-14	2.33

[a] Total thickness.

TABLE IV.—*Concluded.*

Panel	Nickel Thickness, mils	Panel	Nickel Thickness, mils	Panel	Nickel Thickness, mils
No. 15-21	1.47	No. 17-12	1.77	No. 21-21	0.71
No. 15-35	1.39	No. 17-9	1.73	No. 21-24	0.75
No. 15-16	1.33	No. 17-13	1.75	No. 21-15	0.70
No. 15-12	1.30	No. 17-5	1.74	No. 21-12	0.78
No. 15-35	1.39	No. 17-11	1.74	No. 21-34	0.80
No. 52-15	1.43	No. 18-15	1.41	No. 51-21	0.66
No. 41-15	1.42	No. 18-23	1.34	No. 52-21	0.74
No. 21-15	1.53	No. 18-13	1.36	No. 53-21	0.70
No. 11-15	1.51	No. 18-32	1.38	No. 33-21	0.89
No. 23-15	1.55	No. 18-33	1.42	No. 12-21	0.87
No. 15-3	1.48	No. 2C-18	2.30	No. 22-16	2.56
No. 15-2	1.47	No. 3C-18	2.50	No. 22-26	2.62
No. 15-4	1.49	No. 4C-18	2.16	No. 22-24	2.76
No. 15-13	1.51	No. 2B-18	1.62	No. 22-33	2.64
No. 15-7	1.52	No. 2A-18	1.74	No. 22-35	2.80
No. 16-14	1.39	No. 19-24	1.50	No. 31-22	3.0+
No. 16-26	1.40	No. 19-36	1.52	No. 41-22	2.68
No. 16-31	1.52	No. 19-35	1.47	No. 22-22	2.56
No. 16-33	1.39	No. 19-13	1.60	No. 42-22	2.80
No. 16-23	1.38	No. 19-14	1.59	No. 33-22	3.0+
No. 31-16	1.74	No. 2A-19	2.72	No. 23-35	1.43
No. 51-16	1.62	No. 5B-19	2.20	No. 23-21	1.47
No. 32-16	1.64	No. 3B-19	2.80	No. 23-24	1.51
No. 42-16	1.62	No. 4A-19	2.33	No. 23-16	1.55
No. 43-16	1.68			No. 23-36	1.55
No. 16-2	1.64	No. 20-12	1.49	No. 21-23	2.02
No. 16-3	1.64	No. 20-23	1.42	No. 32-23	1.88
No. 16-6	1.62	No. 20-35	1.37	No. 53-23	1.57
No. 16-15	1.61	No. 20-33	1.46	No. 41-23	1.62
No. 16-13	1.57	No. 20-15	1.46	No. 12-23	1.88
No. 17-25	1.48	No. 23-20	2.56		
No. 17-34	1.49	No. 31-20	2.52		
No. 17-21	1.34	No. 41-20	2.41		
No. 17-33	1.35	No. 32-20	2.68		
No. 17-15	1.37	No. 53-20	2.17		
No. 42-17	1.57	No. 20B-14	1.60		
No. 32-17	1.80	No. 20B-31	1.57		
No. 33-17	1.78	No. 20B-34	1.47		
No. 51-17	1.53	No. 20B-22	1.41		
No. 53-17	1.85	No. 20B-26	1.54		

TABLE V.—PLATING THICKNESS—MAGNE-GAGE PANELS EXPOSED AT KURE BEACH, N. C.

Panel	Nickel Thickness, mils	Panel	Nickel Thickness, mils	Panel	Nickel Thickness, mils
No. 1-23	1.40	No. 5-11	...	No. 10-12	1.34
No. 1-32	1.35	No. 5-15	...	No. 10-21	1.32
No. 1-12	1.35	No. 5-22	...	No. 10-23	1.40
No. 1-15	1.57	No. 5-31	...	No. 10-26	1.51
No. 1-16	1.59	No. 5-31	...	No. 10-32	1.38
No. 11-1	1.64	No. 31-5	...	No. 21-10	1.59
No. 21-1	1.68	No. 41-5	...	No. 31-10	2.16
No. 31-1	1.60	No. 51-5	...	No. 42-10	1.96
No. 52-1	1.82	No. 13-5	...	No. 23-10	2.29
No. 43-1	1.60	No. 53-5	...	No. 33-10	2.29
No. 1A-23	1.40	No. 6-15	1.48	No. 11-11	1.35
No. 1A-26	1.43	No. 6-16	1.51	No. 11-13	1.39
No. 1A-21	1.40	No. 6-24	1.47	No. 11-24	1.43
No. 1A-14	1.51	No. 6-31	1.41	No. 11-33	1.47
No. 1A-32	1.47	No. 6-32	1.38	No. 11-36	1.45
No. 1B-11	1.50	No. 31-6	1.88	No. 41-11	1.53
No. 1B-14	1.51	No. 41-6	1.52	No. 22-11	1.90
No. 1B-24	1.53	No. 12-6	2.13	No. 42-11	1.80
No. 1B-32	1.47	No. 13-6	2.04	No. 23-11	2.09
No. 1B-33	1.41	No. 23-6	2.13	No. 53-11	1.47
No. 2-15	1.43[a]	No. 7-12	1.64[a]	No. 12-14	1.52
No. 2-21	1.44[a]	No. 7-14	1.77[a]	No. 12-16	1.50
No. 2-35	1.57[a]	No. 7-25	1.62[a]	No. 12-25	1.37
No. 2-32	1.41[a]	No. 7-34	1.70[a]	No. 12-24	1.37
No. 2-25	1.50[a]	No. 7-16	1.68[a]	No. 12-23	1.42
No. 31-2	1.84[a]	No. 11-7	1.93[a]	No. 41-12	1.78
No. 22-2	1.87[a]	No. 21-7	1.93[a]	No. 51-12	1.58
No. 23-2	1.78[a]	No. 12-7	1.69[a]	No. 12-12	2.02
No. 13-2	1.79[a]	No. 42-7	1.48[a]	No. 22-12	1.63
No. 32-2	1.84[a]	No. 53-7	1.44[a]	No. 23-12	2.09
No. 3-14	1.47	No. 8-24	1.64	No. 13-12	1.49
No. 3-16	1.40	No. 8-25	1.56	No. 13-24	1.50
No. 3-21	1.36	No. 8-36	1.55	No. 13-23	1.37
No. 3-24	1.40	No. 8-32	1.54	No. 13-32	1.41
No. 3-25	1.37	No. 8-33	1.58	No. 13-33	1.49
No. 11-3	1.99	No. 41-8	1.62	No. 11-13	1.60
No. 41-3	1.53	No. 51-8	1.74	No. 21-13	2.15
No. 32-3	2.20	No. 12-8	2.08	No. 32-13	2.09
No. 13-3	2.10	No. 32-8	2.06	No. 52-13	1.88
No. 53-3	1.64	No. 43-8	1.77	No. 33-13	1.85
No. 4-11	...	No. 9-13	1.36	No. 14-11	1.51
No. 4-14	...	No. 9-22	1.38	No. 14-12	1.42
No. 4-25	...	No. 9-26	1.41	No. 14-21	1.37
No. 4-36	...	No. 9-31	1.37	No. 14-23	1.33
No. 4-35	...	No. 9-36	1.41	No. 14-34	1.46
No. 31-4	...	No. 31-9	2.16	No. 21-14	2.37
No. 51-4	...	No. 41-9	1.80	No. 22-14	2.20
No. 12-4	...	No. 52-9	1.82	No. 32-14	2.24
No. 13-4	...	No. 23-9	2.20	No. 42-14	2.40
No. 53-4	...	No. 43-9	1.80	No. 53-14	2.33

[a] Total thickness.

TABLE V—*Concluded.*

Panel	Nickel Thickness, mils	Panel	Nickel Thickness, mils	Panel	Nickel Thickness, mils
No. 15-13	1.39	No. 18-12	1.43	No. 22-14	2.64
No. 15-26	1.33	No. 18-16	1.41	No. 22-21	2.47
No. 15-24	1.38	No. 18-24	1.40	No. 22-25	2.48
No. 15-31	1.61	No. 18-34	1.56	No. 22-36	2.88
No. 15-32	1.43	No. 18-31	1.44	No. 22-34	2.88
No. 31-15	1.47	No. 4A-18	2.24	No. 11-22	3.0+
No. 12-15	1.40	No. 3B-18	1.58	No. 21-22	3.0+
No. 33-15	1.55	No. 4B-18	2.02	No. 32-22	3.0+
No. 43-15	1.51	No. 1C-18	2.20	No. 23-22	3.0+
No. 53-15	1.53	No. 5C-18	1.62	No. 53-22	2.33
No. 15-5	1.52	No. 19-11	1.53	No. 23-11	1.39
No. 15-6	1.52	No. 19-16	1.57	No. 23-15	1.45
No. 15-8	1.49	No. 19-23	1.41	No. 23-25	1.50
No. 15-9	1.54	No. 19-26	1.48	No. 23-31	1.37
No. 15-14	1.50	No. 19-39	1.50	No. 23-33	1.42
No. 16-11	1.33	No. 1A-19	2.68	No. AMIT	1.60
No. 16-12	1.37	No. 5A-19	2.13	No. AM2	1.66
No. 16-13	1.35	No. 2B-19	2.46	No. AM3	1.68
No. 16-24	1.39	No. 1C-19	2.56	No. AM4	1.59
No. 16-25	1.35			No. AM5	1.70
No. 11-16	1.53	No. 20-14	1.56	No. AM6	1.60
No. 21-16	1.70	No. 20-24	1.42	No. AM7	1.56
No. 22-16	1.60	No. 20-21	1.38	No. AM8	1.52
No. 13-16	1.68	No. 20-31	1.40	No. AM9	1.57
No. 33-16	1.80	No. 20-36	1.43	No. AM10	1.57
No. 16-1	1.62	No. 21-20	2.62	No. AM11	1.50
No. 16-5	1.62	No. 51-20	2.37	No. AM12	1.60
No. 16-9	1.60	No. 42-20	2.32	No. AM13	1.48
No. 16-10	1.60	No. 33-20	2.60	No. AM14	1.60
No. 16-11	1.64	No. 43-20	2.24	No. AM15	1.64
No. 17-13	1.40	No. 20B-12	1.52	No. 31-23	1.88
No. 17-14	1.42	No. 20B-13	1.60	No. 51-23	1.66
No. 17-24	1.33	No. 20B-16	1.64	No. 42-23	1.55
No. 17-31	1.40	No. 20B-21	1.54	No. 13-23	1.95
No. 17-32	1.37	No. 20B-33	1.55	No. 43-23	1.54
No. 11-17	1.72	No. 21-16	0.71		
No. 31-17	1.79	No. 21-23	0.72		
No. 22-17	1.82	No. 21-25	0.77		
No. 13-17	1.67	No. 21-33	0.75		
No. 23-17	1.77	No. 21-35	0.75		
No. 17-3	1.73	No. 11-21	0.92		
No. 17-4	1.69	No. 32-21	0.89		
No. 17-8	1.75	No. 42-21	0.72		
No. 17-10	1.77	No. 23-21	0.91		
No. 17-15	1.79	No. 43-21	0.72		

REPORT OF COMMITTEE B-9

ON

METAL POWDERS AND METAL POWDER PRODUCTS*

Committee B-9 on Metal Powders and Metal Powder Products held one meeting during the year in Detroit, Mich., on April 29, 1963.

The committee sponsored the paper by E. W. Lowe and H. R. Biehl on "The Microstructure of Bronze Sinterings," which was published by the Society as *ASTM STP 323.*

The Advisory Committee met twice during the year and approved the following personnel changes: F. I. Zaleski as chairman of Subcommittee III-A replacing J. I. Farmer; C. Brunswic as chairman of Subcommittee III-B replacing O. W. Reen; and J. F. Kuzmick as chairman of Subcommittee III-E replacing A. S. Doty.

Recommendations were received on the proposed Powder Metallurgy Parts Manufacturers Assn. (PMPMA) Coding System.

The committee noted with pride the ASTM Award of Merit received by F. N. Rhines, chairman of Subcommittee I, at the 1962 Annual Meeting.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1962 Annual Meeting, Committee B-9 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Recommended Practice for:

Determination of Transverse Rupture Strength of Cemented Carbides (B 406 – 63 T).

Revision of Tentative Specifications for:

Copper-Infiltrated Sintered Carbon Steel Structural Parts (B 303 – 61 T), and

Steel Backed Metal Powder Bearing and Bushing Alloys (B 377 – 61 T).

Revision of Tentative Method of Test for:

Green Strength of Compacted Metal Powder Specimens (B 312 – 58 T), and

Compressibility of Metal Powders (B 331 – 61 T).

Revision and Reversion to Tentative of Standard Method of Test for:

Sieve Analysis of Granular Metal Powders (B 214 – 56).

These recommendations were accepted by the Standards Committee on Sept. 6, 1963, and all the new and revised tentatives appear in the 1963 Supplement to *Book of ASTM Standards*, Part 3, except the specifications which appear in Part 2.

RECOMMENDATIONS AT ANNUAL MEETING

Tentatives Continued Without Revision

The committee recommends continuation without revision of the following tentatives:

Tentative Methods of Test for:

Hardness of Sintered Metal Friction Materials (B 347 – 59 T),

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

Density of Sintered Metal Friction Materials (B 376 - 61 T), and

Transverse Rupture Strength of Sintered Metal Friction Materials (B 378 - 61 T).

Tentative Specifications for:

Metal Powder Sintered Bearings (Oil Impregnated) (B 202 - 60 T).

ACTIVITIES OF SUBCOMMITTEES

Seven subcommittees met twice during the year: in New York, N. Y. on Oct. 30, 1962, and in Detroit, Mich. on April 29, 1963. Subcommittee III-D had an additional meeting in Detroit on June 19, 1963. Subcommittee III-E did not meet during the year.

Subcommittee I on Nomenclature and Technical Data (F. N. Rhines, chairman). —A task group headed by P. V. Schneider is conducting a feasibility study on devising a standard impact test for sinterings. A task group study of the need for a standard method of test for hardness is continuing. Definitions of twelve new terms are being considered, including new terms adopted by the Metal Powder Industries Federation.

Subcommittee II on Metal Powders (J. B. Haertlein, chairman):

Section A on Base Metal Powders (J. B. Haertlein, chairman) recommended the revision of Methods B 312 and B 331, and the revision and reversion to tentative of Method B 214 as noted earlier in this report. Presently under consideration is a method of test for apparent density of non free-flowing metal powders. Assistance has been given on methods of analytical chemistry of interest in powder metallurgy to Committee E-3 on Chemical Analysis of Metals, Division M, Group I.

Section B on Refractory Metal Powders (W. H. Bleecker, chairman) is refining the details of the photelometric technique as the basis for a method of particle size determination for refractory metal powders. Revisions of the Tentative Method of Test for Average Particle Size of Refractory Metals and Compounds by Fisher Subsieve Sizer (B 330) will be submitted for letter ballot of the subcommittee.

Subcommittee III on Metal Powder Products (H. R. Biehl, chairman):

Section A on Bearings (F. I. Zaleski, chairman) reported publication of *ASTM STP 323*, Microstructure of Bronze Sinterings. A task force headed by H. R. Biehl is continuing its work on the correlation of K factor, machinability, and microstructure of these sintered bronze bearing materials. A task force headed by J. Speck is preparing a specification for low density sintered bronze bearing material, continuing work already accomplished by his task force under W. J. Manella and a tentative specification has been written. The section recommended the revision of Specification B 377 as noted earlier in this report.

Section B on Structural Parts (P. J. Shipe, chairman) has studied industry interest in hardness testing and concluded that while hardness is being used by fabricators for quality control, it is not widely used in specifications. The subcommittee reported letter balloting on revisions of the Tentative Specification for Low and Medium Density Iron, Sintered Metal Powder Structural Parts (B 310) which, when approved, will make obsolete the tentative Specification for High-Density Iron, Sintered Metal Powder Structural Parts (B 309). A specification has been written for iron, copper, carbon sintered metal powder structural parts and submitted to Committee B-9 ballot. Modification of the typical tensile values of Specification B 303 Class C copper infiltrated parts is under discussion.

Section C on Cemented Carbides (C.

Brunswic, chairman) has submitted for Committee B-9 letter ballot a new Tentative Recommended Practice for Hardness Testing of Cemented Carbides. The subcommittee reported pending letter ballot of a tentative recommended practice for determination of electrical resistivity of cemented carbides, an addenda to Methods E 9, covering procedures for determination of compressive strength of cemented carbides; and a tentative recommended procedure for the tensile testing of cemented carbides. The section prepared the new Tentative Recommended Practice for Determination of Transverse Rupture Strength of Cemented Carbides, as indicated earlier in this report.

Section D on Friction Materials (H. B. Huntress, chairman) reported continued extended discussion of wet and dry dynamometer friction testing with emphasis on definition of test conditions and test variables such as temperature, pressure, and speed in wet dynamometer testing. Diversity of friction testing devices in use continues to be an obstacle. Limited funds for equipment and testing among participating companies have hampered the investigation.

Deliberation on dry friction testing is centered on the Chase friction testing machine and the inertia dynamometer procedure. The subcommittee reported formation of four task groups for further studies in the following areas: (1) dry friction clutch dynamometer testing, (2) wet friction dynamometer testing, (3) friction machine testing, and (4) dry friction brake dynamometer testing.

The section recommended the continuation as tentative of Methods B 347, B 376, and B 378 as indicated earlier in this report.

Section E on Machinable Heavy Metals (J. F. Kuzmick, chairman) is continuing with the development of consumer interest in additional specifications for high-density materials, especially new materials which are coming into use.

Respectfully submitted on behalf of the committee,

JOSEPH L. BONANNO,
Chairman.

G. L. BIBBINS,
Secretary.

REPORT OF JOINT ASTM-ASME COMMITTEE
ON
EFFECT OF TEMPERATURE ON THE PROPERTIES OF METALS

Meetings:

The Joint Committee and its various subcommittess and panels met twice during the past year in conjunction with the annual meetings of ASTM in New York, N. Y., June 26–28, 1962, and of ASME in New York, N. Y., Nov. 26–28, 1962. The Panel on Structural Materials for Airframes and Missiles met in Los Angeles, Calif. on Oct. 4, 1962.

Papers and Technical Sessions:

Two papers concerning the use of austenitic stainless steel at elevated temperatures and representing work sponsored by the Steam Power Panel (Projects SP5 and SP6) were presented at the Power Conference in Baltimore, Md., in September, 1962, and have been scheduled for publication in the ASME *Transactions* in April, 1963.

A Workshop on Hot Corrosion was held on June 25, 1962, under the auspices of the Gas Turbine Panel. This well-attended event provided for a free interchange of information and ideas and was well received.

A 3-session Symposium on Materials for Aircraft, Missiles, and Space Vehicles was held in Los Angeles during the ASTM Fourth Pacific Area Meeting, Oct. 4 and 5, 1962. The papers will be published by ASTM as a special technical publication, *ASTM STP No. 345*.

Membership and Officers:

An election of officers was held at the June, 1962, meeting with the following individuals duly nominated and elected:

Chairman, G. V. Smith.

Vice-Chairman, P. M. Brister.

Secretary, H. C. Cross.

Members of Executive Committee: H. M. Soldan, R. C. Fitzgerald, and J. J. Kanter.

Claude Clark resigned as chairman of the Chemical and Petroleum Panel, and was succeeded by Maynard Northup.

Finances:

The status of funds in the committee's account as of January 31, 1963, is given in Table I.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1962 Annual Meeting, the joint committee presented to ASTM, through the Administrative Committee on Standards, the following recommendations:

New Tentative Method for:

Conducting Drop-Weight Test to Determine Nil-Ductility Transition Temperature of Ferritic Steels (E 208 – 63 T).

New Tentative Recommended Practice for:

Compression Tests of Metallic Materials at Elevated Temperatures with Conventional or Rapid Heating Rates and Strain Rates (E 209 – 63 T).

These new tentatives were accepted by the Standards Committee on April 29, 1963, and are available as separate reprints.

ACTIVITIES OF PANELS

Applied Research Panel (W. Lee Williams, chairman):

Meetings of this panel were held on June 26 and Nov 27, 1962. The status of its activities is summarized as follows:

TABLE I.—STATUS OF FUNDS IN THE ACCOUNT OF THE JOINT COMMITTEE AS OF JANUARY 31, 1963.

Balance on hand, Dec. 31, 1961				$111 076.30
Receipts Jan. 1, 1962 through Jan. 31, 1963				12 068.81
Total				$123 145.11
Disbursements, Jan. 1, 1962 through Jan. 31, 1963				29 265.35
Balance on hand, Jan. 31, 1963				$93 879.76
Disbursement by Projects:				
	Contract Price	*Paid*	*Balance*	
Steam Power Panel:				
SP-5, Use of Austenitic Alloy Steels in Steam Line Service (University of Michigan)	$47 500	$47 494.79	$5.21	
SP-6, Use of Austenitic Stainless Steels in Superheater Tubes (University of Michigan)	60 000	59 990.15	9.85	
Data and Publications Panel:				
DP-11-13, Punch Card Tabulation of Data	9 000	6 239.90	2 760.10	
DP-14, Evaluation of Data for the Boiler and Pressure Vessel Code Committee	7 000	6 511.22	488.78	
Applied Research Panel:				
AR-1, Materials Defects in Thermal Fatigue	30 000	8 359.99	21 640.01	
				$24 903.95
Appropriations:				
Metals Data Council				$25 000.00
Chemical and Petroleum Panel Project				$18 000.00
Obligated				$67 903.95
Estimated unallocated balance, Jan. 31, 1963				25 975.81

Project AR-1: The Role of Surface and Material Defects in Thermal Fatigue.—Under a steering committee comprised of R. M. Curran (chairman), J. Miller and S. S. Manson, final contractual arrangements were completed for a 2-yr program of study at Southern Research Inst. A first progress report has been received concerned primarily with a description of the approach and the equipment, but including also a few preliminary results on Type 304 stainless steel.

Project AR-2: Long-Time Creep Rupture Data.—This project is being carried forward under the guidance of a task group of which Mr. Semchysen is chairman. Included in the activities of this project is a cooperative test program, now under way, with 7 participating laboratories, to conduct 50,000 to 100,000-hr tests at 1100 and 1200 F in the type 304 stainless steel specimen bank material prepared by the Test Methods Panel. Test stresses for these long-time tests were selected on the basis of shorter-time tests conducted at the Naval Engineering Experiment Station.

W. E. Leyda made available to the Panel summary tables representing data reported at the 1960 conference in Düsseldorf on long-life European creep and rupture tests, and Mr. Williams reported the completion for Panel distribution of further translations of papers on long-life tests reported at the 1957 Düsseldorf conference.

Data and Publications Panel (G. V. Smith, chairman):

This panel met on June 26 and on Nov. 27, 1962, and the status of its activities may be summarized as follows:

Project DP-13: Continuing Data Survey and Punch Card Tabulation of Data.—Approximately 125 additional data sheets, principally for austenitic stainless steels, have been transmitted to ASTM headquarters for publication.

Project DP-14. Evaluation of Data for the ASME Boiler and Pressure Vessel Committee.—This activity, taken up in June, 1960, at the request of the Subgroup on Metals Engineering of the ASME Boiler and Pressure Committee, to fulfill a need with respect to the setting of working stresses, has progressed more slowly than had been hoped and has proved more costly than originally envisioned. It has become apparent that a greatly expanded effort at data analysis is required, as contemplated in the planning for a Metals Data Council, discussed below. Since such an expansion is not currently possible in the light of the limited financial resources of the committee, this project has been permitted to lapse.

Project DP-15. Survey of Data on Austenitic Stainless Steels.—Since some years have elapsed since the publication of *ASTM STP No. 124*, covering data on austenitic stainless steels, the Panel has set out to revise this publication, under the direction of a steering committee consisting of W. F. Simmons (chairman), W. E. Leyda, and R. M. Curran.

Gas Turbine Panel (Bruce O. Buckland, chairman):

This panel met on June 26 and Nov. 28, 1962. In this panel, task force and problem appraisal committees are active in the following areas:

(*a*) Creep damage evaluation. There is growing evidence that a substantial fraction of the original rupture life of turbine blades removed from service can sometimes be restored by re-heat treatment.

(*b*) Fatigue damage. A task force has been charged with studying the current status of the problem and with bringing recommendations to the panel.

(*c*) Repair welding. Activity in this area is limited at present to following the work under government contract at Republic Aviation.

(*d*) Evaluation of blade corrosion by a screening test involving the plasma jet.

(*e*) Hot corrosion. The success of a workshop on the subject, sponsored by the panel, has indicated that work in this area should be followed and fostered.

The panel has taken steps to support an effort to establish an industry-wide gas turbine fuel specification.

Low-Temperature Panel (A. L. Tarr, chairman):

Meetings of this panel were held on June 28 and Nov. 28, 1962. The panel has collaborated in the work of establishing recommended practices or test methods for (1) the NRL drop weight test, (2) Young's modulus, and for (3) conductivity at cryogenic temperatures.

Task groups have been established to consider research problems in the following areas: aluminum and its alloys;

stainless steels; ferritic steels; and titanium. Consideration will also be given to evaluating the suitability of these materials for low-temperature service.

The panel has given consideration to supporting research programs to evaluate the properties of cryogenically stretch-formed type 304-L stainless steel for service at cryogenic and higher temperatures.

G. W. Geil resigned as secretary of the panel and S. Epstein accepted appointment to this position.

Petroleum and Chemical Panel (M. S. Northup, chairman):

Meetings of this panel were held on June 27, and Nov. 27, 1962. A major activity of this panel is the determination of the creep and rupture properties of AISI type 310 wrought stainless steel and ACI grade HK40 cast stainless steel at 1800, 1900, and 2000 F for up to 10,000 hr. A cooperative program in conjunction with the Alloy Castings Inst. has been developed and extends work already in progress at Battelle Memorial Inst. under sponsorship of ACI.

Steam Power Panel (P. M. Brister, chairman):

Meetings of the panel were held on June 27 and Nov. 30, 1962. The principal activities of the panel are summarized below:

Project SP-5: Use of Austenitic Stainless Steels in Main Steam Piping.—The work at the University of Michigan, guided by a project committee of which R. H. Caughey is chairman, has been completed and a paper summarizing the work was presented at the September 1962 Power Conference in Baltimore. This paper has been published in ASME *Transactions*, April, 1963. No further work is contemplated.

Project SP-6: Use of Austenitic Stainless Steels in Superheater Tubes.—A paper summarizing the work at the University of Michigan on this project was presented at the Baltimore Power Conference and has been published in ASME *Transactions*, April, 1963. The panel is not recommending further work.

Project SP-7: Material Evaluation.—A task group under the chairmanship of R. F. Gill is interested in listing factors important to evaluation of new materials, or for new applications of existing materials and is endeavoring to prepare an evaluation data sheet of general acceptability. This material will be circulated to other ASTM-ASME groups for comment.

Project SP-8: Effect of Long-Time Service on Elevated Temperature Properties of Tubes and Pipes.—A task group, under the leadership of R. L. Dombey, is interested in evaluating remaining life of materials after using up part of the life in extended service.

Task Group on T-11 Material: As the result of several failures, attributed to overheating, of type T-11 superheater tubes in a power station of the Tennessee Valley Authority, a task group under C. A. Waugaman has surveyed the available data for this material. The data apparently do not support present code stresses.

Panel on Structural Materials for Airframes and Missiles (J. J. Heger, chairman):

This panel met on June 27, 1962, in New York, N. Y. and on Oct. 4, 1962, in Los Angeles. The program and activities of this panel are summarized as follows:

Task Group AM-4 on Refractory Metals.—A draft report prepared by a task group under S. E. Bramer has been made available to the Materials Advisory Board for guidance in the possible revision of MAB Report 176-M on Evaluation Test Methods for Refractory Sheet Materials.

Task Group AM-7 on Thermal Physical Properties in the Range above 500 C.—Consideration of the problems of thermal property measurements at elevated temperatures has indicated the desirability of arranging a symposium on the subject.

Problems Dealing with Supersonic Transport Aircraft.—The materials problems of supersonic transport are of interest, but inasmuch as work in this area is being coordinated by FAA, NASA, and the Air Forces, the panel is not being requested to provide assistance. Liaison with the Government work is provided by R. H. Raring.

Task Group on New Materials.—An ad hoc committee, with R. B. Clapper, chairman, has been charged with suggesting appropriate activities for the panel in the broad area of new materials and new material applications.

Test Methods Panel (J. W. Freeman, chairman):

This panel met on June 27 and Nov. 28, 1962, and has been active in the following areas of standardization:

1. Tentative Method for Static Determination of Young's Modulus at low and elevated temperatures. This method is being processed by a task group under W. F. Simmons.

2. Short-Time Compression Testing. A Recommended Practice for Compression Testing of Metallic Materials with Conventional or Rapid Heating Rates and Strain Rates has been completed under the direction of J. R. Kattus.

3. Compression, Bearing, and Shear Creep Tests. After determining that there is no demand for these practices, no action is being taken, currently.

4. Short-Time Shear Testing. J. F. Boysen was appointed chairman of a task group to prepare a practice for this form of testing. The request for such a practice originated with the Aerospace Industries Assn. of America.

5. Notched Specimen Creep-Rupture Testing. A recommended practice for such testing is being processed by a task group under R. Ritter.

6. Drop Weight Test. Following a request by the Naval Research Laboratory, a task group under P. P. Puzak completed the preparation of a Method for Conducting Drop Weight Test to Determine Nil-Ductility Transition Temperatures of Ferritic Materials.

7. Practice for Determination of Thermal Conductivity. At the request of the Panel on Structural Materials for Air Frames and Missiles, consideration is being given to preparing a practice for measurement of thermal conductivity.

8. The panel is also giving consideration to preparing a recommended practice for creep relaxation testing.

9. ISO Specifications. The panel is seeking to acquaint itself with the work of the International Standards Organization.

10. Test Specimen Bank for Rupture-Test Units. Additional tests have been completed and a statistical analysis of the results has been prepared by W. L. Williams. Plans are proceeding to make available stock of the test bank material to interested testing laboratories.

International Conference on Creep (N. L. Mochel, secretary):

An International Conference under the joint sponsorship of the British Institution of Mechanical Engineers and the ASME and ASTM is scheduled for New York, N. Y., in August, 1963.

The program, now nearing final shape, will include several papers by individuals associated with the ASME-ASTM Joint Committee on Effect of Temperature on the Properties of Metals.

Metals Data Council:

A task group consisting of N. L. Mochel (chairman), H. M. Soldan, P. M. Brister, and M. S. Northup, has been exploring the possibility of establishing a "permanent organization set up on a cooperative basis to serve as a central agency for the generation, collection, and processing of data on the properties of metals, for the general benefit of the engineering community, the public interest, and the National Defense." It is proposed that this activity be financed by annual subscriptions.

Approval of the plan to develop such an activity was given by the ASTM Board of Directors in October, 1962, and the Executive Committee of ASME Council approved in November, 1962, planning action by the Research Department and the Joint Committee.

On Dec. 14, 1962, a special meeting attended by representatives of ASME and ASTM, the members of the task force, and the chairman of the Joint Committee was held in New York, N. Y., to work out details of scope, name, location, financing, and operation of the proposed council. Although agreement was reached on a number of these questions, the discussions brought out the general view that the proposed Council is potentially a very important operation and that time should be provided for further detailed consideration of some of the questions of financing and method of operation.

This report has been submitted to letter ballot of the committee, which consists of 26 members; 21 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

G. V. SMITH,
Chairman.

H. C. CROSS,
Secretary.

REPORT OF ADVISORY COMMITTEE
ON
CORROSION

The Advisory Committee on Corrosion held one meeting during the year on June 26, 1962, in New York, N. Y.

PERSONNEL

The officers recently elected for the ensuing term of two years beginning 1963 are as follows:

Chairman, O. B. Ellis.

First Vice-Chairman, W. H. Ailor.

Second Vice-Chairman, R. B. Saltonstall.

W. S. Garwood has been recommended for reappointment as a member-at-large for a three-year term beginning June, 1963. A new committee representative for Committee A-5 is W. M. Barnum.

STATUS OF ATMOSPHERIC EXPOSURE PROGRAMS

Committee A-5 on Corrosion of Iron and Steel:

Inspections of the six atmospheric exposure programs were made during the year. Reports of the inspections of the aluminum and zinc-coated sheets and the variously coated hardware are appended to the 1963 report of this committee.[1]

Committee B-3 on Corrosion of Nonferrous Metals and Alloys:

The data from the exposure of bolted-plate type galvanic couple specimens of two magnesium alloys in contact with a variety of aluminum, steel, copper, and nickel alloys are now available.[2] It is expected that the specimens remaining at Newark, N. J., State College, Pa., and Kure Beach (80-ft site), N. C., will be removed within the year.

The data from a 2-yr operation of apparatus to determine time-of-wetness of specimens and atmospheric SO_2 content have been collected. A report on these data is being prepared.

Visual inspection of the 77 alloys exposed in 1958 was made this spring and has been reported.[3]

Committee B-4 on Metallic Materials for Thermostats and for Electrical Resistance, Heating, and Contacts:

The 2-yr data from an atmospheric exposure of 360 static contact measurements covering a wide variety of alloys used in electrical and electronic components have been collected. An analysis of these data is being prepared for publication.

Committee B-8 on Electrodeposited Metallic Coatings:

The comparative performance of the type and thickness of chromium deposits over duplex nickel and a variety of copper-nickel coatings is now available (Program No. 5).[4]

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[1] Appendixes I and II, Report of Committee A-5, see pp. 132 and 136.

[2] Appendix I, Report of Committee B-3, see p. 168.

[3] Appendix II, Report of Committee B-3, p. 170.

[4] Appendix I, Report of Committee B-8, see p. 212.

Exposure of 88 specimens from Program Nos. 5 and 7 is being made on trucks operating out of Detroit, Mich. The results of this exposure will determine the future direction of this type of study.

CORROSION RESISTANCE OF ELECTRODEPOSITED COATINGS

Although commercial electroplating began in the 1890's, it was long after the turn of the century that serious effort was made to determine the protective value of these coatings under atmospheric conditions.[5,6]

The first comprehensive study of electroplated coatings utilizing variations in thickness of deposits, electroplating practices, variety and number of coating elements, buffing versus non-buffing, and cleaning procedures was initiated in 1932. This study involving the exposure of over 3000 specimens was a joint project of the American Electroplater's Society, the National Bureau of Standards, and ASTM.[7] A second study followed in 1936 also conducted jointly by the AES, NBS, and ASTM.[8]

In 1941, Committee B-8 on Electrodeposited Metallic Coatings and Related Finishes was organized and all ASTM work in this field became centered in this committee.

Lack of conclusive data on the relative durability of various composite copper-nickel-chromium electrodeposits as protective and decorative coatings on steel led Committee B-8 on Electrodeposited Coatings and Related Finishes in 1947 to undertake a series of studies designated as Program Nos. 1 through 5.[9,10,11] Program No. 7,[1] just started, is designed to study similar coatings on zinc-alloy die-castings.

Program No. 6, exposed in 1962, uses aluminum alloys as the basis metal to study the effects of variations of copper and double-layered nickel as decorative finishes.

Several other atmospheric exposure studies have been conducted by Committee B-8: one of the earlier concerned electroplated lead from fluoborate and sulfamate solutions applied to steel, copper-flashed steel, and copper; and to various coatings such as lead-tin, zinc, hot-dipped lead, and terne.[12]

Polishing and its effect on the protective value of electroplated coatings[13] has also been investigated.

Phosphate coatings over electrodeposited zinc were studied to determine

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[5] E. M. Baker, "Rust Resistance of Nickel Plated Steel," *Journal*, Soc. Automotive Engineering, Vol. 15, p. 300 (1924).

[6] W. M. Phillips, "Further Developments in Low pH Nickel Deposition," Am. Electrochemical Soc., *Technical Proceedings*, Vol. 59, p. 393 (1931).

[7] W. Blum, P. W. C. Strasser, and A. Brenner, "Protective Value of Nickel and Chromium Plating on Steel," *Journal of Research*, Nat. Bureau of Standards, Vol. 13, Sept., 1934, p. 331 (RP 712).

[8] W. Blum and P. W. C. Strasser, "Outdoor Exposure Tests of Electroplated Nickel and Chromium Coatings on Steel and Nonferrous Metals," *Journal of Research*, Nat. Bureau of Standards, Vol. 24, April, 1940, p. 443 (RP 1293).

[9] C. H. Sample, "The Corrosion Behavior and Protective Value of Copper-Nickel-Chromium and Nickel-Chromium Coatings on Steel," Symposium on Properties, Tests, and Performance of Electrodeposited Metallic Coatings, *ASTM STP 197*, Am. Soc. Testing Mats., p. 7 (1956). (Discusses Programs No. 1, 2, and 3.)

[10] "Performance of Decorative Nickel-Chromium Coatings on Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 61, p. 329 (1961). (Program No. 4.)

[11] "Performance of Decorative Nickel-Chromium Coatings," *Proceedings*, Am. Soc. Testing Mats., Vol. 63, p. 215 (1963). (Program No. 4 and 5.)

[12] A. H. Du Rose, "Atmospheric Exposure of Electroplated Lead Coatings on Steel," Symposium on Properties, Tests, and Performance of Electrodeposited Metallic Coatings, *ASTM STP 197*, Am. Soc. Testing Mats., p. 97 (1956).

[13] G. A. Lux and W. Blum, "Methods of Polishing Steel and Their Effects Upon the Protective Value of Electroplated Coatings," *Journal of Research*, Nat. Bureau of Standards, Vol. 34, April, 1945 p. 295 (RP 1645).

whether or not these coatings have merit other than as base coatings for organic finishes.[14]

Chromate conversion films for electroplated zinc coatings on steel were studied both in atmospheric exposures and with accelerated testing procedures[15] in an attempt at correlating the resulting data (Program No. 101).

Cadmium-plated steel panels with supplemental chromate films are also being observed in order to establish standards for the evaluation of the additional protection afforded by these films (Program No. 102).

New atmospheric studies under way show expanding areas of interest of Committee B-8: anodized aluminum panels have been exposed to study effects of weathering; surplus chromium-plated panels from Program Nos. 5 and 7 are being exposed on trucks, tractors, and trailers to evaluate this type of exposure; and electroplated and mechanically plated zinc-coated specimens will be exposed on exposure racks mounted on private automobiles.

Respectfully submitted on behalf of the committee,

C. P. LARRABEE,
Chairman.

S. F. ETRIS,
Secretary.

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[14] A. L. Alexander, "Evaluation of Phosphate Coating over Electrodeposited Zinc," Symposium on Properties, Tests, and Performance of Electrodeposited Metallic Coatings, *ASTM STP 197*, Am. Soc. Testing Mats., p. 66 (1956).

[15] "Exposure and Accelerated Tests of Supplementary Chromate Treatments for Electroplated Zinc Coatings," *Proceedings*, Am. Soc. Testing Mats., Vol. 61, p. 359 (1961).

REPORT OF COMMITTEE C-1
ON
CEMENT*

Committee C-1 on Cement held two meetings during the year: in New York, N. Y., on June 29, 1962, and in Pittsburgh, Pa., on Nov. 27, 1962.

R. R. Litehiser submitted his resignation as chairman, effective at the Annual Meeting, because of his retirement and W. J. Halstead has been designated to fill that office for one year.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee C-1 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] All of the existing standards and tentatives referred to appear in their present form in the 1961 Book of ASTM Standards, Part 4, or the 1962 Supplement thereto.

New Tentative

Tentative Specification for Moist Cabinets and Rooms Used in the Testing of Hydraulic Cements, as appended hereto.[2]

Revision of Tentatives

Tentative Specifications for Portland Blast-Furnace Slag Cement (C 205 – 62 T):

Section 2(d).—At the end of the paragraph, change the value "8" to "8.0."

Table II.—Change the heading of the third column from "Type IS(M)" to read "Type IS(MS)." In the first line delete "(alternate methods)[a]" following "Fineness." Change the fineness requirements for amount retained when wet-sieved on No. 325 sieve from "12" to "12.0" for all four types. Under "Fineness" indent the next line which reads "Specific surface by air permeability apparatus, sq cm per g:" and add a reference to Footnote *a*. Change Footnote *a* to read as follows:

> [a] These limits apply only when specifically requested. Portland blast-furnace slag cement has a lower apparent specific gravity than portland cement, and therefore, when the air permeability apparatus is used, the weight of the sample shall be adjusted so that a firm, hard bed is formed by the compacting process, and the fineness shall be calculated by the appropriate formula using the determined specific gravity.

In the line "Compressive strength, psi" delete reference to Footnote *c*.

Tentative Specifications for Portland-Pozzolan Cement (C 340 – 62 T):

Section 3.—In the last sentence of Paragraphs (*a*) and (*b*), change the expression "15 to 50" to read "15 to 40."

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[2] The new tentative appears in the 1963 Supplement to *Book of ASTM Standards*, Part 4.

Section 9.—After the expression "376 lb net" insert "(Note 4)." Add the following new Note 4 at the end of the section and renumber present Note 4 as Note 5:

NOTE 4.—A 94-lb bag of portland-pozzolan cement has a larger absolute volume than a 94-lb bag of portland cement, which may be taken into consideration in purchasing cement and in proportioning concrete mixtures.

Section 13.—Change Paragraph (*c*) to read: "(*c*) *Fineness by Sieving.*—Method of Test for Fineness of Hydraulic Cement by the No. 325 Sieve (ASTM Designation: C 430)."

In Paragraph (*l*)(*1*), delete the phrase "not less than 70 per cent by weight of CaO and." Substitute the words "on the ignited basis" for the phrase "based on the nonvolatile portion."

Table I.—Change the chemical requirement of sulfur trioxide from "2.5" to read "3.0." In the last line change "loss of ignition" to read "loss on ignition."

Table II.—Change the indicated parts of the entries pertaining to fineness, soundness, time of setting, and mortar expansion to read as shown in the accompanying Table I, including a new Footnote *a*. Reletter present Footnotes *a*, *b*, and *c* as *b*, *c*, and *d*.

TABLE I.—PHYSICAL REQUIREMENTS.
(Changes in Table II of Specifications C 340)

	Type IP	Type IP-A
Fineness:		
Amount retained when wet-sieved on No. 325 ($44\text{-}\mu$) sieve, max, per cent	12.0	12.0
Specific surface by air permeability apparatus, sq cm per g:[a]		
Average value, min	2800	2800
Minimum value, any one sample	2600	2600
Soundness:		
Autoclave expansion or contraction, max, per cent[b]	0.50	0.50
Time of setting (alternate methods):[c]		
Mortar expansion:[d]		

[a] These limits apply only when specifically requested. Portland-pozzolan cement has a lower apparent specific gravity than portland cement, and, therefore, when the air permeability apparatus is used, the weight of the sample shall be adjusted so that a firm, hard bed is formed by the compacting process and the fineness shall be calculated by the appropriate formula, using the determined specific gravity.

Tentative Method of Test for Flexural Strength of Hydraulic Cement Mortars (C 348 – 61 T):

Section 11(a).—Insert a new second and third sentence to read as follows:

All test specimens for a given test age shall be broken within the permissible tolerance prescribed below:

Test Age	Permissible Tolerance
24 hours	±½ hr
3 days	±1 hr
7 days	±3 hr
28 days	±12 hr

When the portions of prisms are to be tested as modified cubes (Method C 349), the prisms shall be tested early enough so that the modified cubes are also broken within the above tolerances.

Tentative Specifications for Slag Cement (C 358 - 62 T):

Section 6.—Delete Paragraph (*b*) and Note 2. Reletter Paragraph (*c*) as (*b*).

Section 9.—Change the third sentence to read: "A bag shall contain 80 lb net." Delete Note 3.

Table I.—Change to read as follows:

Sulfur trioxide (SO_3), max, per cent	4.0
Sulfide sulfur (S), max, per cent	2.0
Insoluble residue, max, per cent	1.0
Loss on ignition, max, per cent	4.0

Table II.—Change the limits on autoclave expansion from "0.5" to "0.50" and the limits on initial set from "45" to "60" in both columns.

Tentative Method of Test for Potential Sulfate Resistance of Portland Cement (C 452 - 60 T):

Title.—Change to read: "Tentative Method of Test for Potential Expansion of Portland Cement Mortars Exposed to Sulfate."

Section 1.—Change to read as follows:

1. This method of test, which is applicable only to portland cements, covers a procedure for the determination of the expansion of mortar bars made from a mixture of portland cement and gypsum in such proportions that the mixture has an SO_3 content of 7.0 per cent by weight. This method of test is intended primarily for use by those interested in research on methods for determining the potential sulfate resistance of portland cement.

Section 3.—Replace the existing Paragraphs (*a*) and (*b*) with a new Section 3 to read "3. The temperature of the air and the relative humidities of the laboratory and moist closet shall conform to the requirements of Section 3 of Method C 109."

Sections 4 and 5.—Delete the existing Sections 4 and 5, and add a new Section 4 to read as follows and renumber present Section 6 as Section 5:

Materials

4. (*a*) The graded sand for making the test specimens shall conform to the requirements of Section 4 of Method C 109.

(*b*) The gypsum used for addition to the portland cement shall be high grade natural gypsum with 100 per cent passing the No. 100 (149-μ) sieve, at least 94 per cent passing the No. 200 (74-μ) sieve and at least 90 per cent passing the No. 325 (44-μ) sieve (Notes 2 and 3). Calculate the percentage of cement and gypsum required to provide a mixture containing 7.0 per cent SO_3 by weight as follows:

$$\text{Cement, per cent} = \frac{g - 7.0}{g - c} \times 100$$

$$\text{Gypsum, per cent} = \frac{7.0 - c}{g - c} \times 100$$

where:

c = SO_3 content of the portland cement, per cent,

g = SO_3 content of the gypsum, per cent, and

7.0 = SO_3 content of the cement-gypsum mixture, per cent.

NOTE 2.—Terra Alba No. 1, available from the U. S. Gypsum Co., Southard, Okla. plant, meets these requirements.

NOTE 3.—If the SO_3 content of the gypsum is unknown, the gypsum shall be analyzed for SO_3 content using the Method of Chemical Analysis of Gypsum and Gypsum Products (ASTM Designation: C 471).

Section 7.—Renumber as Section 6 and change to read as follows:

6. The interior surfaces of the mold shall be covered with a plastic tape or thinly covered with lard oil (Note 4). The gage studs shall then be set, care being taken to keep them clean and free of oil. Beeswax or paraffin heated to the melting point, or a mixture of three parts of paraffin to five parts of rosin by weight, heated between 110 and 120 C (230 and 248 F), shall be applied to the outside contact lines of the molds to make the molds watertight.

Note 2.—Renumber as Note 4 and change to read: "NOTE 4.—Lard oil is susceptible to self ignition. Special precautions should be used in the disposition of waste rags containing the lard oil."

Section 8.—Renumber as Section 7.

Change the first sentence in Paragraph (*a*) to read: "The quantities of dry materials required for each batch shall be 400 g (cement plus gypsum) and 1000 g sand."

Change Paragraph (*b*) to read: "The mortar shall be mixed in accordance with the procedure described in Section 6 of Method C 305, except in step 2 first add the gypsum to the water, then start the mixer and mix at slow speed (140 ± 5 rpm) for 15 sec."

Section 9.—Renumber as Section 8 and change the first sentence to read: "Immediately upon completion of the flow test, return the mortar to the bowl, scrape down into the batch any mortar on the side of the bowl, and remix at the medium speed (285 ± 10 rpm) for 15 sec."

Section 10.—Renumber as Section 9. In Paragraph (*a*), delete the portion of the second sentence which reads: "which shall be made at the age of 24 hr ± 15 min from the time the cement and water are mixed together."

In Paragraph (*b*), insert the word "horizontally" after the word "stored" in line 3, and add a new third sentence to read as follows: "The specimens shall be covered with at least $\frac{1}{2}$ in. of water."

Section 11.—Renumber as Section 10 and add a reference to Note 5 at the end of Paragraph (*b*).

Section 12.—Renumber as Section 11. In the second sentence, change the value "0.002" to "0.004", and delete the words "percentage points" and the reference to Note 3.

Note 3.—Renumber as Note 5 and relocate following the new Section 10.

Tentative Specifications for Processing Additions for Use in the Manufacture of Portland Cement (C 465 – 62 T):

Section 2(e)(2).—Change to read: "(*2*) The time of setting of cement containing the addition shall not vary from the time of setting of the respective blank cement by more than 1 hr or 50 per cent, which ever is the lesser."

Tentative Revision of Standard

Standard Method of Test for Normal Consistency of Hydraulic Cement (C 187 – 58):

Section 4(a).—Change to read as follows:

> 4. (*a*) *Preparation of Cement Paste.*—Mix 500 g of cement with a measured quantity of clean water following the procedure prescribed in Section 5 of the Method for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency (ASTM Designation: C 305).

Revision of Standard,
Immediate Adoption

(requires nine-tenths affirmative vote)

Standard Specifications for Masonry Cement (C 91 – 60):

Section 13.—Change to read: "13. The residue on the No. 325 (44-μ) sieve shall be determined in accordance with the Method of Test for Fineness of Hydraulic Cement by the No. 325 Sieve (ASTM Designation: C 430)."

Section 20(b).—Change to read as follows:

> (*b*) *Mixing of Mortars.*—The mortar shall be mixed in accordance with Section 6 of Method C 305. If the mortar has the correct flow, another portion of the mortar shall be used for the determination of entrained air. (See Section 25(*b*)). After determination of flow and air content, return all of the mortar to the mixing bowl and remix for 15 sec at medium speed. Cubes shall then be made as prescribed in Section 21(*a*).

Section 24(d).—Change to read: "(*d*) *Tapping Stick* made of maple wood and having a diameter of $\frac{5}{8}$ in. and a length of 6 in."

Section 25(b).—Replace the first two sentences with the following sentence:

"When the quantity of mixing water has been found that produces a flow of 110 ± 5, using another portion of mortar, immediately determine the weight of 400 ml of mortar." Delete the word "flat" in the present sixth sentence. Add the following new sentence at the end of the section: "Then if cubes are to be made, the operations described in Sections 20(*b*) and 21(*a*) should be started immediately."

Section 26.—In the explanation of S_2 change the value "2.648" to "2.65."

Section 27(*a*).—Change the fifth sentence to read: "The bore of the stopcock shall have a diameter of 4 ± 0.5 mm, and the connecting glass tubing shall have a minimum inside diameter of 4 mm."

Standard Method of Test for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. Cube Specimens) (C 109 – 58):

Section 12(*a*).—Insert a new second sentence and tabular material to read as follows: "All test specimens for a given test age shall be broken within the permissible tolerance prescribed as follows:"

Test Age	Permissible Tolerance
24 hours	±½ hr
3 days	±1 hr
7 days	±3 hr
28 days	±12 hr

Standard Specifications for Portland Cement (C 150 – 62):

Table I.—Change the loss on ignition for Type IV from "2.3" to read "2.5." Add as a new requirement for Type V: "Tetracalcium aluminoferrite plus twice the tricalcium aluminate[c] ($4\ CaO \cdot Al_2O_3 \cdot Fe_2O_3 + 2(3\ CaO \cdot Al_2O_3)$, or solid solution ($4\ CaO \cdot Al_2O_3 \cdot Fe_2O_3 + 2\ CaO \cdot Fe_2O_3$), as applicable, max, per cent . . . 20.0."

Change the equations in Footnote (*c*) to read as follows:

Tricalcium silicate = (4.071 × per cent CaO) – (7.600 × per cent SiO_2) – (6.718 × per cent Al_2O_3) – (1.430 × per cent Fe_2O_3) – (2.852 × per cent SO_3).

Dicalcium silicate = (2.867 × per cent SiO_2) – (0.7544 × per cent C_3S).

Tricalcium aluminate = (2.650 × per cent Al_2O_3) – (1.692 × per cent Fe_2O_3).

Tricalcium silicate = (4.071 × per cent CaO) – (7.600 × per cent SiO_2) – (4.479 × per cent Al_2O_3) – (2.859 × per cent Fe_2O_3) – (2.852 × per cent SO_3).

Table II.—In the tabular portion, delete all entries pertaining to tensile strength[3] and add the requirement "False set, final penetration, min, per cent,[f] . . . 50" to apply to all types of cement. Add a new Footnote *f* to read as follows:

[f] This requirement applies only when specifically requested.

Section 10.—Add a new Paragraph (*k*) to read: "(*k*) *False Set.*—Method of Test for False Set of Portland Cement (Paste Method) (ASTM Designation: C 451)."

Standard Methods of Test for Autoclave Expansion of Portland Cement (C 151 – 60):

Section 2(*e*).—Replace the present first sentence with three sentences worded as follows: "The autoclave shall consist of a high-pressure steam vessel provided with a thermometer well. The autoclave shall be equipped with an automatic pressure control and a rupture disk with a bursting pressure of 350 psi ± 5 per cent. In locations where the use of a rupture disk is not permitted, the autoclave shall be equipped with a safety valve."

Notes 2 and 3.—Renumber as Notes 4

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[3] This part of the committee's recommendations failed of approval.—Ed.

and 5 and insert new Notes 2 and 3 immediately after Section 2(*e*) to read as follows:

NOTE 2.—The temperature of the rupture disk will be at some value between room temperature and 420 F. The disk material should have a tensile strength relatively insensitive to temperature in the above range.

NOTE 3.—The disk should be electro-chemically compatible with the pipe leading to it and to its holder.

Notes 4 to 6.—Renumber as Notes 6 to 8.

Section 5(a).—Change the second sentence to read: "The autoclave shall contain enough water, at an initial temperature of 20 to 27.5 C (68 to 81.5 F), to maintain an atmosphere of saturated steam vapor during the entire test."

Standard Specifications for Air-Entraining Portland Cement (C 175 – 61):

Section 3.—At the end of the section, reference a new Footnote 4 to read: "[4]Compliance with the requirements of this specification does not necessarily insure that the desired air content will be obtained in concrete."

Section 10.—Add a new Paragraph (*j*) to read "*False Set.*—Method of Test for False Set of Portland Cement (Paste Method) (ASTM Designation: C 451)."

Table I.—Change certain of the equations in Footnote *a* to read as follows:

Tricalcium silicate = (4.071 × per cent CaO) − (7.600 × per cent SiO_2) − (6.718 × per cent Al_2O_3) − (1.430 × per cent Fe_2O_3) − (2.852 × per cent SO_3).

Dicalcium silicate = (2.867 × per cent SiO_2) − (0.7544 × per cent C_3S).

Tricalcium aluminate = (2.650 × per cent Al_2O_3) − (1.692 × per cent Fe_2O_3).

Tricalcium silicate = (4.071 × per cent CaO) − (7.600 × per cent SiO_2) − (4.479 × per cent Al_2O_3) − (2.859 × per cent Fe_2O_3) − (2.852 × per cent SO_3).

Table II.—In the tabular portion, add the requirement "False set, final penetration, min, per cent,[e] . . . 50" to apply to all types of cement. Add a new Footnote *e* to read: "[e]This requirement applies only when specifically requested."

Standard Method of Test for Tensile Strength of Hydraulic Cement Mortars (C 190 – 59):

Section 9(a).—Insert a new second sentence and tabular material to read as follows: "All test specimens for a given test age shall be broken within the permissible tolerance prescribed in the following table:"

Test Age	Permissible Tolerance
24 hours	±½ hr
3 days	±1 hr
7 days	±3 hr
28 days	±12 hr

Tentatives Continued Without Revision

Tentative Specifications for:

Air-Entraining Additions for Use in the Manufacture of Air-Entraining Portland Cement (**C 226 – 61 T**), and

Flow Table for Use in Tests of Hydraulic Cement (**C 230 – 61 T**).

Tentative Methods of Test for:

Chemical Analysis of Portland Cement (**C 114 – 61 T**),

Sampling Hydraulic Cement (**C 183 – 61 T**),

Calcium Sulfate in Hydrated Portland Cement Mortar (**C 265 – 58 T**),

Time of Setting of Hydraulic Cement by Gillmore Needles (**C 266 – 58 T**),

Mechanical Mixing of Hydraulic Cement

Pastes and Mortars of Plastic Consistency (**C 305 – 59 T**),
Compressive Strength of Hydraulic Cement Mortars (Using Portions of Prisms Broken in Flexure) (**C 349 – 59 T**),
False Set of Portland Cement (Mortar Method) (**C 359 – 61 T**), and
Fineness of Hydraulic Cement by the No. 325 Sieve (**C 430 – 60 T**)

Editorial Changes

Standard Method of Test for Normal Consistency of Hydraulic Cement (**C 187 – 58**):

Section 2(d).—In the tabulation of weights and dimensions, change the words "weight of plunger" to "weight of movable rod" and the words "diameter of larger end of plunger" to "diameter of plunger end of rod."

Section 5.—Change to read: "The amount of water required for normal consistency shall be calculated to the nearest 0.1 per cent and shall be reported to the nearest 0.5 per cent of the weight of the dry cement."

This report has been submitted to letter ballot of the committee, which consists of 90 voting members; 71 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. R. LITEHISER,
Chairman.

W. J. MCCOY,
Secretary.

REPORT OF COMMITTEE C-3
ON
CHEMICAL-RESISTANT NONMETALLIC MATERIALS*

Committee C-3 on Chemical-Resistant Nonmetallic Materials held two meetings during the year: on Oct. 3 to 5, 1962, in Williamsburg, Va., and on Feb. 20 to 22, 1963, in Haddonfield, N. J.

The committee currently consists of 27 members, of whom 10 are classified as producers, 9 as consumers, and 8 as general interest members, with two consulting members.

During the year, 10 representatives were approved by the committee as new members (some representing multiple membership) and 4 resigned because of changes in professional interest.

Because of the change in emphasis of the committee's activities during the past few years and to improve the efficiency of operation, the four subcommittees previously responsible for silicate, resin, sulfur, and hydraulic mortars were combined into a single subcommittee titled S-1 on Chemical-Resistant Mortars, which will be responsible for all specifications in the assigned area.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee C-3 requests approval by the Society at the Annual Meeting of the following recommendations. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] All of the existing tentatives and standards referred to appear in their present form in the 1961 Book of ASTM Standards, Part 4, or the 1962 Supplement.

Revision of Tentatives

Tentative Method of Test for Absorption and Apparent Porosity of Chemical-Resistant Mortars (C 413 – 58 T):

Section 3.—Change Paragraph (*d*) to read as follows including the two new notes:

(*d*) *Specimen Molds,* for right cylinders 1 in. in diameter and 1 in. high. The molds shall consist of either 1 in. thick flat plastic sheet in which 1 in. diameter, smooth-sided holes have been cut, and to the bottom of which a $\frac{1}{4}$ in. thick flat plastic sheet without holes is attached by means of screws or bolts, or shall consist of sections of round plastic tubing or pipe 1 in. in inside diameter and 1 in. long, having sufficient wall thickness to be rigid and retain dimensional stability during the molding operation, and a $\frac{1}{4}$ in. thick flat plastic sheet on which one open end of each section can be rested (Note 1).

NOTE 1.—For use with sulfur mortars a piece of flat plastic sheet at least $\frac{1}{8}$ in. thick containing a rectangular hole $\frac{1}{2}$ in. long by $\frac{3}{32}$ in. wide with slightly tapered sides is required.

NOTE 2.—The material from which the mold is constructed must be chemically inert and have anti-stick properties. Polyethylene, polypropylene, trifluorochloroethylene polymer (Kel-F), and tetrafluoroethylene (Teflon) have been found satisfactory. Because of their superior heat resistance, only Kel-F or Teflon should be used with sulfur mortars.

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Delete Paragraph (*e*), and reletter Paragraph (*f*) as (*e*).

Sections 6, 7, and 8.—Delete present sections and add new Sections 6, 7, and 8 to read as follows:

Test Specimens

6. (*a*) The test specimens shall be cast right cylinders, 1 ± $\frac{1}{16}$ in. in diameter by 1 ± $\frac{1}{16}$ in. high, with flat, smooth faces normal to the axis of the cylinder, prepared in molds as described in Section 3(*d*) and employing no release agent in the mold.

(*b*) The number of specimens required is dependent upon the number of test solutions to be employed, the number of different temperatures at which testing is performed, and the frequency of test intervals. The test specimens shall consist of sets of two cylinders for one solution at a single temperature and for each test interval. In addition, one set of two specimens shall be available for test immediately following the conditioning period and other sets of two, equivalent to the number of test temperatures, for test after aging in air at the test temperature for the total test period. The total number of specimens required shall be calculated as follows:

$$N = 2(S \times T \times I) + 2T + 2$$

where:

N = number of specimens,
S = number of solutions,
T = number of test temperatures, and
I = number of test intervals.

Preparation of Specimens

7. (*a*) The method of preparation of specimens is dependent upon the type of mortar to be tested. They shall be prepared as described in Paragraphs (*b*) to (*d*). If the faces of a specimen are not flat, smooth, and normal to the cylinder axis, they may be sanded, ground, or machined to specification. Care should be taken that the frictional heat developed during such operations does not damage the specimen.

(*b*) *Resin-Type Mortars.*—Mix in the proper portions and in the manner specified by the manufacturer of the materials. Place in the mold with a spatula, taking care to ensure complete filling of the mold cavity without entrapment of air. Scrape off the excess mortar even with the face of the mold, making the exposed surface as smooth and even as possible. Permit the mortar to remain in the mold until it has set sufficiently to allow removal without danger of deformation or breakage.

(*c*) *Silicate-Type Mortars.*—Follow the same procedure described in Paragraph (*b*), leaving the specimens in the mold for 18 to 24 hr after molding.

(*d*) *Sulfur Mortars.*—Melt a minimum of 2 lb of the material in a suitable container in not over 1 hr and hold at a temperature of 265 to 290 F for at least 15 min with constant agitation. The stirring shall be such as to lift the aggregate without beating air into the melt. Place the piece of plastic sheet containing the rectangular hole, beveled side down, over the open face of the mold with the hole centered on the face. On top of the piece of plastic sheet and surrounding the rectangular hole place a section of plastic tubing or pipe 1 in. in diameter by 1 in. high. Pour the melted mortar through the rectangular hole into the mold, and continue to pour until the section of tubing or pipe is completely filled. The excess mortar contained in the section of tubing or pipe acts as a reservoir to compensate for shrinkage of the mortar during cooling. Allow the specimen to remain in the mold until it has completely solidified. Upon removal, file, grind, or sand off the surface flush, removing the excess material remaining at the pouring gate.

(*e*) *Hydraulic Mortars.*—Mix the mortar, employing the same proportions of water, cement, and aggregates as are to be used in service. The aggregates shall be of the type and origin of the aggregates to be used in service. Cast the specimens in accordance with the procedure described in Paragraph (*b*).

Conditioning Test Specimens

8. Age the cast specimens for a period of seven days in air at a temperature of 73.4 ± 4 F (23 ± 2 C), the seven days to include time in the mold.

Tentative Method of Test for Chemical Resistance of Mortars (C 267 – 62 T):

Section 6(*c*).—Add a new Note to this section to read as follows:

NOTE.—In reference to silicate and hydraulic mortars, the testing laboratory shall first prepare and test in accordance with Section 9 (*c*), sufficient specimens (a minimum of 10) and attain, before running chemical resistance tests, a standard deviation not greater than 10 per cent based on the following formula:

$$\sigma = \sqrt{\frac{\Sigma x^2}{n} - \bar{x}^2}$$

where:

σ = standard deviation in pounds,

x = result of individual determination,

$\bar{x}$ = average = $\frac{\Sigma x}{n}$, and

n = number of samples.

Revision of Standards
Immediate Adoption

(requires nine-tenths affirmative vote)

Standard Recommended Practice for Use of Chemically Setting Silicate-Type Chemical-Resistant Mortars (C 397 – 62):

Section 3.—Add a new Paragraph (*g*) to read as follows:

(*g*) When silicate or silica mortars or both are to be exposed to temperatures below 50 F or above 80 F during mixing, application, or setting, special precautions are required. The manufacturer should be consulted for specific recommendations.

Standard Recommended Practice for Use of Resin-Type Chemical-Resistant Mortars (C 399 – 60):

New Section.—Add a new Section 12 to read as follows, including the tabular material:

12. A general guide for the use of resin-type mortars at elevated temperatures is given below. The ratings are for dry heat in air only. Where used otherwise, chemical resistance at the temperature in question determined by the Method of Test for Chemical Resistance of Mortars (ASTM Designation: C 267), and specific manufacturer's recommendations should be taken into consideration.

DRY HEAT LIMITS IN AIR OF RESIN-TYPE MORTARS.

Resin	Dry Heat, max, deg Fahr[a]
Phenolic	300
Furan	300
Epoxy:	
ambient temperature systems	160
heat-cured systems	300
Polyester	225

[a] For higher temperature uses than above, the suppliers shall be consulted for specific recommendation.

Standard Specification for Resin-Type Chemical-Resistant Mortars (C 395 – 60)

Table I.—Change to read as shown in the accompanying Table I.[2]

Tentatives Continued
Without Revision

Tentative Methods of Test for:

Flexural Strength of Chemically Setting Silicate-Type Chemical-Resistant Mortars (**C 453 – 60 T**), to allow more time for experience with the Method.

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[2] Table I is not included in this report but appears in the revised standard; see 1964 *Book of ASTM Standards*, Part 12.

Working and Initial Setting Times of Chemically Setting Silicate-Type Chemical-Resistant Mortars (**C 414 – 61 T**), to allow further consideration of "Set Time."

This report has been submitted to letter ballot of the committee, which consists of 27 members; 24 members returned ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. H. BURTON,
Chairman.

W. H. PEARLSON,
Secretary.

REPORT OF COMMITTEE C-4

ON

CLAY PIPE*

Committee C-4 on Clay Pipe had one meeting during the year: on June 26, 1962, in New York, N. Y.

The committee consists of 27 members, of whom 11 are classified as producers, 13 as consumers, and 3 as general interest members.

At the June meeting, the committee adopted a memorial to William Eber Robinson, a member since 1932, who died on May 13, 1962.

At the 1962 Annual Meeting, Robert G. Scott received the ASTM Award of Merit.

Tentatives Continued Without Revision

The committee recommends that the following tentatives be continued without revision:

Tentative Recommended Practice for:

Installing Vitrified Clay Sewer Pipe (**C 12 - 58 T**).

Tentative Methods of Testing:

Clay Pipe (**C 301 - 60 T**).

Tentative Specifications for:

Standard Strength Clay Sewer Pipe (**C 13 - 57 T**),
Extra Strength Clay Pipe (**C 200 - 59 T**),
Standard Strength Unglazed Clay Pipe (**C 261 - 60 T**),
Extra Strength Unglazed Clay Pipe (**C 278 - 60 T**)
Vitrified Clay Pipe Joints Using Materials Having Resilient Properties (**C 425 - 60 T**),
Standard Strength Ceramic Glazed Clay Pipe (**C 462 - 60 T**), and
Extra Strength Ceramic Glazed Clay Pipe (**C 463 - 60 T**).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Methods of Tests and Specifications is continuing its study of the revision of terminology in Method C 301 and the revision of "Scope" of Specification C 425 - 60 T.

This report has been submitted to letter ballot of the committee, which consists of 27 members; 22 members returned their ballot, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

C. R. VELZY,
Chairman.

ROBERT G. SCOTT,
Secretary.

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

REPORT OF COMMITTEE C-5
ON
MANUFACTURED CARBON AND GRAPHITE PRODUCTS

Committee C-5 on Manufactured Carbon and Graphite Products was organized in Cleveland, Ohio, on May 21, 1963.

The scope of the committee is:

> *Scope.*—The promotion of knowledge, stimulation of research, and the development of methods of test, specifications, and nomenclature for manufactured carbon and graphite products exclusive of those assigned to other ASTM committees. These activities will be correlated with those of other related committees of ASTM and other organizations (specifically ASTM Committees C-6, C-8, D-1, D-2, D-24, and D-28).

The officers elected for the ensuing term of two years are as follows:

Chairman, James Woodburn, Jr.
Vice-Chairman, Bruce L. Bailey.
Secretary, William W. Palmquist.

The committee consists of 18 members, of whom 10 are classified as producers, 7 as consumers, and 1 as a general interest member.

Seven subcommittees were formed and their activities are as follows:

Subcommittee I, Executive (James Woodburn, Jr., chairman) is surveying the interests in the carbon and graphite field to determine if new subcommittees should be formed to cover these activities.

Subcommittee II on Nomenclature (Frank Rusinko, Jr., chairman) is assembling a list of terms of specific interest to the carbon and graphite field.

Subcommittee III on Property Measurements (W. J. Spry, chairman) is exploring existing chemical and physical test methods to assist in determining the subcommittee program. Task groups were formed to explore room temperature test procedures, elevated temperature tests, and special test methods.

Subcommittee IV on Mechanical Graphite (Harry Tankus, chairman) was organized on Dec. 12, 1962, in Chicago, Ill. This subcommittee covers the development of test methods for graphite mechanical seals and is working on a standard method for tension testing of these materials.

Subcommittee V on Nuclear Grade Graphite (Robert Meyer, chairman) was organized under the auspices of Committee C-21 on Whitewares, on Feb. 10, 1960, in Philadelphia, Pa., under the chairmanship of W. C. Riley. A draft specification based on three grades of graphite has been developed. Drafts of six physical test methods have been circulated as well as methods for moisture and ash determination. Colorimetric methods for determining small quantities of silicon, iron, calcium, aluminum, titanium, and vanadium are being studied.

Subcommittee VI on Impregnated Graphite Pipe (John Reys, chairman) will define the sizes and grades of graphite pipe and determine the temperature and pressure limitations of use. The work will be mostly physical property measurement and will include permeability methods.

Subcommittee VII on Electrical Contacts (Paul Smisko, chairman) is exploring areas of electrical applications of possible interest to the committee.

Respectfully submitted on behalf of the committee,

James Woodburn, Jr.
Chairman.

William W. Palmquist,
Secretary.

REPORT OF COMMITTEE C-6
ON
PYROLYTIC MATERIALS

Committee C-6 on Pyrolytic Materials held its organizational meeting in Philadelphia, Pa., on Feb. 25, 1963. The scope for the new committee is:

> The promotion of knowledge, stimulation of research, and the development of methods of test, specifications, and nomenclature for pyrolytic materials.
>
> These activities will be coordinated with those of other related committees of ASTM and other organizations.

The committee consists of 11 members, of whom 2 are classified as producers, 5 as consumers, and 4 as general interest members. There are 6 consulting members.

At that time, two subcommittees were formed: Subcommittee I on Nomenclature, and Subcommittee II on Specifications and Methods of Testing.

The officers elected for the ensuing term of two years are as follows:

Chairman, J. D. McClelland.
Vice-Chairman, E. F. Keon.
Secretary, J. Persh.
Membership Secretary, E. G. Stevens.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature (G. R. Hammeter, chairman).—The first work of this subcommittee will be to develop a definition for the word "pyrolytic" as specifically requested by the Board of Directors.

Subcommittee II on Specifications and Methods of Testing (W. H. Smith and H. E. Martens, co-chairmen).—The preliminary specification for pyrolytic graphite was developed at the subcommittee meeting held on June 21, 1963, in Pittsburgh, Pa. Draft methods for room-temperature bend testing, high- and low-temperature tension testing, and thermal conductivity tests were discussed.

Ad Hoc Committee on Information Exchange (E. F. Keon, chairman).—A symposium on pyrolytic materials is being organized for presentation at the next meeting of the committee to be held in April, 1964, in Los Angeles, Calif.

Respectfully submitted on behalf of the committee,

J. D. McCLELLAND,
Chairman.

J. PERSH,
Secretary.

REPORT OF COMMITTEE C-8
ON
REFRACTORIES*

Committee C-8 on Refractories held two meetings during the year: the 102nd meeting at Bedford Springs, Pa., on Oct. 4, 1962, and the 103rd meeting at Pittsburgh, Pa., on Feb. 15, 1963.

The committee consists of 51 members, of whom 24 are classified as producers, 20 as consumers, and 7 as general interest members.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1962 Annual Meeting, Committee C-8 presented to the Society through the Administrative Committee on Standards tentative revisions of the Standard Definitions of Terms Relating to Refractories (**C 71 – 62**).

The tentative revisions were accepted by the Standards Committee on March 21, 1963, and they appear in the 1963 *Manual of ASTM Standards on Refractory Materials.*

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee C-8 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] The existing tentatives referred to appear in their present form in the 1961 Book of ASTM Standards, Part 5, or the 1962 Supplement thereto.

Tentative Revisions of Standards

Standard Methods of Test for Crushing Strength and Modulus of Rupture of Insulating Fire Brick at Room Temperature (C 93 – 54):

Section 3.—Change to read as follows:

3. The test specimens shall consist of 10 pieces measuring approximately 4½ by 4½ by 2½ in. or 3 in. taken from 10 different brick.

Standard Classification of Silica Refractory Brick (C 416 – 60):

Section 1.—Change to read as follows:

1. This classification is limited to silica brick meeting the following requirements:

Alumina (Al_2O_3) content of less than 1.50 per cent.

Titania (TiO_2) content of less than 0.20 per cent.

Iron oxide (Fe_2O_3) content of less than 2.50 per cent.

Calcium oxide (CaO) content of less than 4.00 per cent.

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Average modulus of rupture of not less than 500 psi.

Adoption of Tentatives as Standard Without Revision

Tentative Classification of:

Steel Pouring Pit Refractories (**C 435 – 61 T**), and
Refractory Granular Dolomite (**C 468 – 61 T**).

Adoption of Tentative Revision as Standard

Standard Definitions of Terms Relating to Refractories (**C 71 – 62**):

Adopt as standard the definitions for "Calcined Refractory Dolomite" and "Dead-Burned Refractory Dolomite," as issued June, 1956, and modified June, 1958, and June, 1959.

This report has been submitted to letter ballot of the committee, which consists of 51 members; 37 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. S. DEBENHAM,
Chairman.

F. W. SCHROEDER,
Secretary.

REPORT OF COMMITTEE C-9
ON
CONCRETE AND CONCRETE AGGREGATES*

Committee C-9 on Concrete and Concrete Aggregates held two meetings during the year: on June 26, 1962, during the Annual Meeting of the Society in New York, N. Y., and on Nov. 30, 1962, at Pittsburgh, Pa.

The committee adopted memorials to: Morton O. Withey, an Honorary Member and member of the committee since 1914, who died on Dec. 11, 1961, and to Frederic B. Lysle, a member of the committee since 1920, who died on Aug. 28, 1961.

A citation was presented to Stanton Walker who was elected to Honorary Membership of the committee at the June, 1962, meeting.

The Sanford E. Thompson Award, established by the committee in 1938, will be made this year to Delmar L. Bloem, Richard D. Gaynor, and John R. Wilson, of the National Ready-Mixed Concrete Assn., and the National Sand and Gravel Association, for their paper entitled "Testing Uniformity of Large Batches of Concrete," which was presented at the 64th Annual Meeting of the Society, June, 1961, and was published in the 1961 Proceedings.

A report on "The Inspection of Concrete Testing Laboratories," prepared jointly for Committees C-1 and C-9 by the joint subcommittee on the Cement and Concrete Reference Laboratory, is appended.[2]

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee C-9 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] All of the existing tentatives and standards referred to in this report appear in their present form in the 1961 Book of ASTM Standards, Part 4, and the 1962 Supplement thereto.

New Tentatives

as appended hereto.[3]

Tentative Method of Test for:

Creep of Concrete in Compression, and

Securing, Preparing, and Testing Specimens From Hardened Lightweight Insulating Concrete for Compressive Strength.

Revision of Tentatives

Tentative Method of Test for Potential Alkali Reactivity of Cement-Aggregate

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*** Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.**

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[2] See p. 281.

[3] The new tentatives appear in the 1963 Supplement to *Book of ASTM Standards*, Part 4.

Combinations (Mortar Bar Method) (C 227 - 61 T):

Section 1.—Change to read as follows renumbering subsequent notes accordingly:

1. This method covers determination of the susceptibility of cement-aggregate combinations to expansive reactions involving the alkalies (sodium and potassium) by measurement of the linear expansion developed by the combination in mortar bars during storage under prescribed conditions of test.

NOTE 1.—Alkalies participating in the expansive reactions usually are derived from the cement; under some circumstances they may be derived from other constituents of the concrete or from external sources. Two types of alkali reactivity of aggregates are recognized: (*1*) an alkali-silica reaction involving certain siliceous rocks, minerals, and artificial glasses and (*2*) an alkali-carbonate reaction involving dolomite in certain calcitic dolomites and dolomitic limestones. The method is not recommended as a means to detect the latter reaction.

Section 4(b).—Delete the last sentence and replace it with: "The portions retained on the various sieves shall then be dried and, unless used immediately, each such portion shall be stored individually in a clean container provided with a tight-fitting cover."

Section 5(c).—Change the second sentence to read as follows:

The quantities of dry materials to be mixed at one time in the batch of mortar for making two specimens shall be 300 g of cement and 675 g of aggregate made up by recombining the portions retained on the various sieves (Section 4(*b*)) in the grading prescribed in Table II or of aggregate in a grading meeting the requirements of the specifications for the project.

Section 10.—In lines 6 and 11, replace "batch of mortar" with "cement-aggregate combination."

Section 11.—Add a new Section 11 entitled "Interpretation of Results" to read as follows:

11. (*a*) Data correlating the results of tests performed using this method with performance of cement-aggregate combinations in concrete in service, results of petrographic examination of aggregates and results of tests for potential reactivity of aggregates by chemical methods have been published in Section 16 of the Method of Test for Potential Reactivity of Aggregates (Chemical Method) (ASTM Designation: C 289) and should be consulted in connection with the use of results of tests performed using this method as the basis for conclusions and recommendations concerning the use of cement-aggregate combinations in concrete.

(*b*) The results of tests performed using this method furnish information on the likelihood that a cement-aggregate combination is potentially capable of harmful alkali reactivity with consequent deleterious expansion of concrete. Criteria to determine potential deleterious alkali reactivity of cement-aggregate combinations from the results of this method of test have been given in Note 3, Paragraph (*3*) of the Specifications for Concrete Aggregates (ASTM Designation: C 33); they are essentially as follows: Cement-aggregate combinations which, when tested by this method, show expansions greater than 0.10 per cent at 6 months usually should be considered capable of harmful reactivity. When 6-month results are not available, combinations should be considered potentially capable of harmful alkali reactivity if they show expansions greater than 0.05 per cent at 3 months.

(*c*) Insignificant expansion may result when potentially deleteriously reactive siliceous rocks are present in comparatively high proportion even when a high-alkali cement is used, probably because the alkali-silica reaction products are characterized by an alkali to silica ratio that is so low as to minimize uptake of water and swelling. Dolomitic aggregates that are deleteriously affected by the alkali-carbonate reaction when employed as coarse aggregate in concrete may not produce notable expansion in this method of test. Also, significant expansion may occur rarely in the test for reasons other than alkali-aggregate reaction, particularly the presence of sulfates in the aggregate that produce a sulfate attack upon the cement paste, ferrous sulfides (pyrite, marcasite, or pyrrhotite) that oxidize and hydrate with the release of sulfate, and materials such as free lime (CaO) or free magnesia (MgO) in the cement or aggregate that progressively hydrate and carbonate.

(*d*) When expansions in excess of 0.10 per cent at 6 months (or in excess of 0.05 at 3 months) are shown in results of tests performed using this method, it is strongly recommended that supplementary information be developed to confirm that the expansion is actually due to alkali reactivity. Sources of such supplementary information include: (*1*) petrographic examination of the aggregate to determine if known reactive constituents are present; (*2*) examination of the specimens after test as provided for in Section 8 to identify the products of alkali reactivity; and (*3*) tests of the aggregate for potential reactivity by chemical methods, (Method C 289).

(*e*) When it has been concluded from the results of tests performed using this method and supplementary information as outlined above that a given cement-aggregate combination should be considered potentially deleteriously reactive, additional studies may be appropriate to develop information on the potential reactivity of other combinations containing the same cement with other aggregates, the same aggregate with other cements, or the same cement-aggregate combination with a mineral admixture.

Tentative Method of Test for Potential Reactivity of Aggregates (Chemical Method) (C 289 – 61 T):

Section 3.—Change the title to read "Reagents and Reagent Containers."

In the last line of Paragraph (*c*), change "bottle" to "containers" and add "(Note 1)" at the end of the paragraph.

Add a new Note 1 to read as follows renumbering subsequent Notes accordingly:

NOTE 1.—In selection of the container, care should be taken to assure that the reagent will not be modified by reaction with the material composing the container, including pigments or other additives, or by transpiration of phases through the walls of the container. Containers with wall thickness not less than 0.020 in. and composed of high-density polyethylene meeting the requirements of the Specifications for Polyethylene Molding and Extrusion Materials (ASTM Designation: D 1248) for materials of Type III, Class A, are suitable.

In Paragraphs (*d*), (*e*), (*f*), (*i*), and (*l*) add a new last sentence to read: "Store the solution in a chemically resistant glass or suitable plastic container (Note 1)."

In Paragraph (*g*), add a new last sentence to read: "Store the solution in a lead, wax, paraffined-paper, or suitable plastic container (Note 1)."

In Paragraph (*h*), change the last sentence to read: "Store the solution in a chemically resistant glass or suitable plastic container (Note 1)."

In Paragraph (*j*), change the second sentence to read: "Store the solution in a paraffin-coated, chemically resistant glass bottle, a hard rubber bottle, or a plastic container of a type that has been shown not to affect the composition or concentration of the solution (Note 1)."

In Paragraph (*k*), delete the last sentence and replace it with the following:

Store the solution in a paraffin-coated, chemically resistant glass bottle, a hard rubber bottle, or a plastic container of a type that has been shown to not affect the composition or concentration of the solution (Note 1). Protect the dry reagent and solution from contamination by carbon dioxide.

Tentative Method of Test for Effectiveness of Mineral Admixtures in Preventing Excessive Expansion of Concrete Due to the Alkali-Aggregate Reaction (C 441 – 62 T):

Section 3(a).—Change to read as follows, retaining the present footnote:

3. (*a*) *Pyrex Glass.*—Crushed Pyrex glass in a grading as follows shall be used as the aggregate in the preparation of the mortar bars:

Sieve Size		Percentage by Weight
Passing	Retained on	
No. 4...... (4760-μ)	No. 8...... (2380-μ)	10
No. 8...... (2380-μ)	No. 16..... (1190-μ)	25
No. 16..... (1190-μ)	No. 30..... (590-μ)	25
No. 30..... (595-μ)	No. 50..... (297-μ)	25
No. 50..... (297-μ)	No. 100.... (149-μ)	15

After the Pyrex glass has been separated into the various sieve sizes, each size shall be washed with a water spray over the sieve to remove adhering dust and fine particles from the aggregate. The portions retained on the various sieves shall then be dried and, unless used immediately, each such portion shall be stored individually in a clean container provided with a tight-fitting cover.

Section 4.—Change the last part of the first sentence of Paragraph (*a*) to read "... cement and 900 g of Pyrex glass aggregate made by recombining the portions retained on the various sieves in the grading prescribed in Section 3(*a*) (Note 2)."

Change the last part of the first sentence of Paragraph (*b*) to read "... and 900 g of Pyrex glass aggregate made by recombining the portions retained on the various sieves in the grading prescribed in Section 3(*a*) (Note 3)."

Section 11.—In lines 4 and 10, change "batch of mortar" to read "cement-aggregate combination."

Tentative Method of Test for Measuring Mortar-Making Properties of Fine Aggregate (C 87 – 62 T):

Revise as appended hereto.[4]

Tentative Method of Test for Lightweight Pieces in Aggregate (C 123 – 57 T):

Section 5.—In the second sentence of Paragraph (*a*) after the words "to the nearest 0.1 g," insert the clause, "and bring this material to a saturated-surface-dry condition (Note 3) by means of the procedure specified in Section 3 of the Method of Test for Specific Gravity and Absorption of Fine Aggregate (ASTM Designation: C 128)."

At the end of this paragraph, add the following note:

NOTE 3.—If the absorption as determined by Method C 128 is known, the fine aggregate may be prepared for test by adding to a known weight of dry sand the amount of water it will absorb, mixing thoroughly, and permitting the sand to stand in a covered pan for 30 min before use.

In the second sentence of Paragraph (*b*) after the words "to the nearest 1g," insert the clause, "and bring to a saturated - surface - dry condition by means of the procedure specified in Section 4 of the Method of Test for Specific Gravity and Absorption of Coarse Aggregate (ASTM Designation: C 127)."

At the end of Section 5, add the following Note 4, referenced to the end of both Paragraphs (*a*) and (*b*):

NOTE 4.—Normally the discrepancy between oven-dry weight and saturated-surface-dry weight of the decanted particles will not significantly affect the calculated percentage of lightweight pieces. If a more precise determination is required, the decanted particles shall be dried to substantially constant weight at 230 ± 9 F (110 ± 5 C) to determine the value of W_1 used for the calculation in Section 6.

Tentative Specifications for Raw or Calcined Natural Pozzolans for Use as Admixtures in Portland Cement Concrete (C 402 – 62 T):

Table II.—In line 3 change the mean particle diameter limit from "9.00" to "9.0."

Section 8.—In line 8, delete "as follows" and replace with "as indicated below. The natural pozzolan shall be used in lieu of fly ash in applicable tests under Method C 311."

In Note 7, change "(*X*—*Y*)" to "(*Y*—*X*)".

Change the first sentence of Paragraph (*j*) to read "Determine the reduction of mortar expansion in accordance with the Method of Test for Effectiveness of Mineral Admixtures in Preventing Excessive Expansion of Concrete Due to the Alkali-Aggregate Reaction (ASTM Designation: C 441)."

Change the first sentence of Paragraph (*k*) to read "Determine the mortar expansion in accordance with the requirements of Method C 441 for the job mixture (see especially Sections 4(*c*) and 9(*b*) of Method C 441)."

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[4] The revised tentative appears in the 1963 Supplement to *Book of ASTM Standards*, Part 4.

Tentative Method of Test for Resistance of Concrete Specimens to Rapid Freezing and Thawing in Water (C 290 – 61 T):

Note 2.—Add the following two sentences to Note 2:

Experience has indicated that ice or water pressure, during freezing tests, particularly in equipment that uses air rather than a liquid as the heat transfer medium, can cause excessive damage to rigid metal containers, and possibly to the specimens therein. Results of tests during which bulging or other distortion of containers occurs should be interpreted with caution.

Tentative Method of Test for Resistance of Concrete Specimens to Slow Freezing and Thawing in Water or Brine (C 292 – 61 T):

Section 5(b).—Add a new last sentence to read: "Results of tests during which bulging or other distortion of the containers occurs should be interpreted with caution."

Tentative Method of Testing Air-Entraining Admixtures for Concrete (C 233 – 60 T):

Section 2(a).—Delete the last sentence and replace it with: "If a blend of cements is used, it shall be a combination which produces an air content of less than 10 per cent when tested in accordance with the Method of Test for Air Content of Hydraulic Cement Mortar (ASTM Designation: C 185) (Note 1)."

Section 2(c).—In the first sentence, delete the words "from which specimens will be made for test for resistance to freezing and thawing as specified in Section 6(*b*)."

Section 2(d).—Change "specimen" to "specimens" at the end of this Paragraph.

Section 3(a)(2).—Add a new Note 2 to read as follows:

NOTE 2.—Values in Table 6 of ACI Recommended Practice 613-54 are intended to assure workable mixtures with the least favorable combinations of aggregate likely to be used. It is suggested, therefore, that for a closer approximation of the proportions required for this test, the values selected from Table 6 be increased by about 7 per cent for the first trial mixture.

Section 3(a)(3).—Delete and add a new Item (*3*) to read as follows:

(*3*) The air content used in the computation of proportions for all concrete shall be 5.5 per cent except where the admixture under test is for use in specific work (see Section 2(*d*)). In this case the air content used in selecting proportions shall be the median of the range to be permitted in the work.

TABLE I.—TYPES AND MINIMUM NUMBER OF SPECIMENS AND TESTS.
(Revision of Table I of Method 233)

Test	Types of Specimens[a]	Test Ages	Minimum Number of Specimens	
			Per Batch	Total
Consistency (slump)	1	1	1	6
Air content	1	1	1	6
Bleeding	1	1	1	6
Compressive strength	1	5	5	30
Flexural strength	1	5	5	30
Freezing and thawing	1	1	2[b]	12
Bond to steel	2	1	2	12
Volume change	1	5	1	6[c]

[a] See Sections 5 and 6.
[b] Specimens for duplicate tests from each batch.
[c] Same specimens used for tests at all ages.

Section 3(b).—Delete and add a new Paragraph (*b*) toread as follows:

(*b*) *Conditions.*—Concrete mixtures shall be prepared both with the air-entraining admixture under test and with the reference admixture. The admixtures shall be added in the amounts necessary to produce the design air content prescribed in Section 3(*a*)(*3*) ± 0.5 per cent of the volume of concrete.

Section 4.—Delete and add a new Section 4 to read as follows:

4. (*a*) *Procedure.*—Concrete shall be machine-mixed in accordance with Section 4(*c*) of ASTM Method C 192. The concrete shall be mixed for 2 min after all materials have been introduced

into the mixer, allowed to rest in the mixer for 3 min, remixed for 1 min, and then discharged.

(*b*) *Number of Batches and Specimens.*—At least three separate batches of concrete with the reference admixture and three with the admixture under test shall be prepared. On any one day, the same number of batches shall be made with each admixture. Specimens of the types and in the numbers listed in Table I shall be made and tested from each batch. For compressive and flexural strengths, a specimen from each batch shall be tested at each specified age.

Table I.—Change to read as shown in the accompanying Table I.

Section 6.—Delete and add a new Section 6 entitled "Tests of Hardened Concrete," to read as follows:

6. Specimens shall be made and tested in accordance with the following methods, and the minimum number of tests shall be as prescribed in Table I. Specimens shall be examined visually before they are tested and any specimen found to be manifestly faulty by such examination shall be discarded without being tested. Specimens shall also be examined visually after testing, and the results obtained from any found manifestly faulty by such examination shall be discarded. Should more than one specimen representing a given test at a given age be found manifestly faulty either before or after testing, the entire test shall be discarded and repeated. The test results reported shall be the average of the individual test results of the specimens tested or, in the event that one specimen or one result has been discarded, the average of the remaining specimens.

(*a*) *Compressive Strength.*—Test specimens shall be made and cured in accordance with ASTM Method C 192 and tested in accordance with the Method of Test for Compressive Strength of Molded Concrete Cylinders (ASTM Designation: C 39). Specimens shall be tested at ages of 3, 7, and 28 days, 6 months, and 1 year (Note 3).

(*b*) *Flexural Strength.*—Test specimens shall be made and cured in accordance with ASTM Method C 192 and tested in accordance with the Method of Test for Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading) (ASTM Designation: C 78). Specimens shall be tested at ages of 3, 7, and 28 days, 6 months, and 1 year (Notes 3 and 4).

(*c*) *Resistance to Freezing and Thawing.*—Test specimens shall consist of molded beams not less than 3 nor more than 5 in. in width and depth and not less than 16 in. in length, tested in accordance with the Method of Test for Resistance of Concrete Specimens to Rapid Freezing and Thawing in Water (ASTM Designation: C 290). Specimens shall be tested at the age of 14 days.

(*d*) *Bond to Steel.*—Test specimens shall consist of cubes and prisms of concrete with vertically and horizontally embedded steel, prepared and tested in accordance with the Method of Test for Comparing Concretes on the Basis of the Bond Developed with Reinforcing Steel (ASTM Designation: C 234). Specimens shall be tested at an age of 28 days (Note 4). The strength of the bond shall be evaluated in terms of the nominal average bond stress measured at five equal intervals of slip at the loaded end taken from the bond stress curves, over the range of 0 to 0.01-in. slip. If the maximum slip for any specimen or specimens in the sets being compared is less than 0.01 in., then the upper limit of the range over which the comparisons are to be made shall be the lowest maximum slip in the group.

(*e*) *Volume Change.*—Test specimens shall consist of molded prisms made and tested in accordance with the Method of Test for Volume Change of Cement Mortar and Concrete (ASTM Designation: C 157). Specimens shall be tested at ages of 3, 7, and 28 days, 6 months, and 1 year (Notes 3 and 4).

NOTE 3.—When tests are conducted with materials representative of those proposed for use in specific work in accordance with Section 2(*d*), and if the results of the tests are required in a period of time which will not permit curing of specimens to ages of 6 months and 1 year, the tests at those ages as required by Section 6(*a*), (*b*), and (*c*) may be waived.

NOTE 4.—Applicable only when specifically required by the purchaser for use in structures where flexural strength, bond strength, or volume change may be of critical importance.

Section 7.—Delete this section.

Section 8.—Renumber as Section 7. In Item (*1*) change "7" to "6". In Item (*3*), delete the words "the material used as". In Item (*7*), change the numbers "1", "2", and "7" to "3", "4", and "6", respectively.

Tentative Specifications for Air-Entraining Admixtures for Concrete (C 260 – 60 T):

Section 3.—Change Paragraphs (*a*), (*b*), (*c*), (*e*), and (*f*) to read as follows:

(*a*) *Bleeding.*—The bleeding of concrete made with the admixture under test shall not exceed that of concrete made with the reference admixture by more than 2 percentage points, the bleeding being computed as a percentage of the net amount of mixing water in each concrete. The net mixing water is the water in excess of that present as absorbed water in the aggregates.

(*b*) *Compressive Strength.*—The compressive strength at any test age of concrete containing the admixture under test shall be not less than 90 per cent of that of similar concrete containing the reference admixture at the same test age.

(*c*) *Flexural Strength.*—The flexural strength at any test age of concrete containing the admixture under test shall be not less than 90 per cent of that of similar concrete containing the reference admixture at the same test age (Note).

(*e*) *Bond Strength.*—The bond strength at the age of 28 days of concrete containing the admixture under test shall be not less than 90 per cent of that of similar concrete containing the reference admixture at the same age (Note).

(*f*) *Volume Change.*—The volume change on drying of concrete containing the admixture under test, expressed as a percentage change in length, shall be not more than 0.010 greater than that of similar concrete containing the reference admixture at ages of 28 days, 6 months, and 1 year (Note).

Tentative Specifications for Fly Ash for Use as an Admixture in Portland Cement Concrete (C 350 – 60 T):

Section 1.—Change Notes 1 and 2 to read as follows:

NOTE 1.—These specifications contemplate use of fly ash as an admixture in combination with portland cement (ASTM Designation: C 150), air-entraining portland cement (ASTM Designation: C 175), portland blast furnace slag cement (ASTM Designation: C 205), or portland-pozzolan cement in which fly ash is the pozzolanic addition (ASTM Designation: C 340). The user should recognize that replacement of a portion of any of these cements by fly ash may reduce early strength, and partial replacement of blended cements by fly ash will in most cases reduce early strength and may reduce resistance of concrete to the effects of freezing and thawing. Care should be exercised to ensure strength and freezing and thawing resistance adequate for the contemplated use of the concrete.

NOTE 2.—Fly ash may tend to reduce the air content of concrete. Hence, if a fly ash is added to any concrete for which entrainment of air is specified, provision should be made to assure that the specified air content is maintained, such as by use of additional air-entraining admixture or use of an air-entraining admixture in combination with air-entraining cement.

Section 5.—In line 8, delete "as follows" and add "as indicated below. Cement of the type proposed for use in the work, preferably from the mill proposed as the source of the cement, shall be used in all tests requiring use of hydraulic cement."

In Paragraph (*g*), change reference to Section 8(*j*) of Specification C 402 to Section 8(*k*).

Tentative Specifications for Chemical Admixtures for Concrete (C 494 – 62 T):

Section 7.—Delete present Paragraph (*b*) and add new paragraphs (*b*), (*c*), and (*d*) to read as follows:

(*b*) *Liquid Admixtures.*—The entire quantity of a lot considered to have been produced or handled in such a manner as to be well blended may be represented by a single sample of at least 1 gal in volume. Such a sample may be secured directly from bulk storage or from a single container representing the lot.

The average quality of not more than 10,000 gal of admixture from different lots, or from a single lot of doubtful uniformity, may be represented by a composite sample. Such a sample shall be the result of compositing a minimum of three samples, each having a volume of at least 1 qt and representing not more than 550 gal or fraction thereof. Samples for compositing shall be taken from different locations, which shall be well distributed throughout the quantity to be represented. The contents of drums from which samples are to be taken shall be agitated thoroughly immediately prior to sampling. Admixtures in bulk storage tanks shall be sampled by obtaining equal quantities of the admixture from the upper, middle, and lower levels by means of drain cocks in the sides of the tanks or a weighted sampling container fitted with a stopper that can be removed after the container is lowered to the desired depth. The contents of the tanks shall be agitated immediately prior to the sampling operation. Samples for compositing shall be throughly mixed and at least 1 gal of the resulting mixture shall be taken to provide the sample for test.

Samples shall be packaged in impermeable, airtight containers.

(*c*) *Non-Liquid Admixtures.*—The entire quantity of a lot considered to have been produced or handled in such a manner as to be well blended may be represented by a single sample weighing at least 10 lb. Such a sample may be secured directly from bulk storage or from a single package representing the lot.

The average quality of not more than 50 tons of admixture from different lots, or from a single lot of doubtful uniformity, may be represented by a composite sample. Such a sample shall be the result of compositing a minimum of four samples, each weighing at least 5 lb and representing not more than 5 tons or fraction thereof. Samples for compositing shall be taken from different locations, which shall be well distributed throughout the quantity to be represented. These samples shall be thoroughly mixed and at least 10 lb of the resulting mixture shall be taken to provide the composite sample for test.

Samples of packaged admixtures shall be taken by means of a tube sampler as described in the Methods of Sampling Hydraulic Cement (ASTM Designation: C 183).

Samples shall be packaged in moisture-proof, airtight containers.

(*d*) Samples shall be thoroughly mixed before testing to assure uniformity. When recommended by the manufacturer, the entire sample of a non-liquid admixture shall be dissolved in water prior to testing.

Tentative Method of Test for Time of Setting of Concrete Mixtures by Penetration Resistance (C 403 – 62 T):

Section 1(a).—Change to read as follows:

1. (*a*) This method provides a procedure for determining the time of setting of concrete with slump greater than zero by testing mortar sieved from the concrete mixture. The method is suitable for use only when tests of the mortar fraction of the concrete will provide the information required. Since the hardening of concrete is a gradual process, any definition of time of setting must necessarily be arbitrary. The temperature of storage of specimens employed in this test is to be selected by the user. Times of initial and final setting of concrete are determined in accordance with this method on the basis of a rate of hardening test made by means of penetration resistance needles on mortar sieved from the concrete mixture.

Section 3(a).—Change to read as follows:

3. (*a*) *Containers for Mortar Specimens.*—Rigid, watertight, non-absorptive, non-oiled containers, either cylindrical or rectangular in cross-section. The minimum lateral dimension shall be 6 in. and the height at least 6 in. (Note 1).

Sections 4 and 5.—Change to read as follows:

4. (*a*) From the concrete mixture under test select a representative sample of concrete of sufficient volume to provide enough mortar to fill the test container, or containers, to a depth of at least 5½ in.

(*b*) Remove essentially all of the mortar from the sample of concrete by sieving it through a No. 4 sieve (4.76 mm)[5] on to a non-absorptive surface.

(*c*) Thoroughly remix the mortar by hand methods on the nonabsorptive surface and place it in the container, or containers, and compact by vibration or rodding; rod the specimen by means of the tamping rod held so as to penetrate the mortar with the round end. If the specimen is compacted by vibration, limit the vibration time to that necessary to produce an approximately level surface after compaction. If the specimen is compacted by rodding, rod the mortar once for each square inch of top-surface area of the specimen, and distribute the strokes uniformly over the cross-section of the specimen. After completion of vibration or rodding, tap the sides of the specimen container lightly with a tamping rod to close voids left by the vibrator or tamping rod and to further level the surface of the specimen. Upon completion of specimen preparation, the mortar surface shall be at least ½ in. below the top edge of the container to provide space for collection and removal of bleeding water and to avoid contact between the mortar surface and the protective covering specified in Section 5.

5. Store and maintain the specimens at the desired temperature of test. To prevent excessive evaporation of moisture, keep the specimens covered with a suitable material such as damp burlap or a tight-fitting, water-impermeable cover for the duration of the test, except when bleeding water is being removed or penetration tests are being made. The specimens shall be shielded from the sun.

Tentative Revision of Standard

Standard Method of Test for Compressive Strength of Molded Concrete Cylinders (C 39 – 61):

Section 2.—Delete the word "normally" in the present third sentence. Add a new third sentence to read as follows:

There shall be sufficient vertical clearance between the testing heads and lateral clearance between the components of the frame to permit insertion of circular proving rings of appropriate capacity and the necessary bearing blocks, so that the open face of the ring will be approximately parallel with the front of the machine.

Add a new paragraph to this section to read as follows:

Each dial on (hydraulically operated) compression machines used in concrete strength tests shall be provided with a graduated scale which is at least 25 in. in length (corresponds to the usable arc of 10-in. diameter circle) and divided into not less than 400 equal parts. The scale shall be provided with a graduation line equal to zero and so numbered. The dial pointer shall be of sufficient length to reach the graduation marks and the width of the leading end shall be not greater than the clear distance between the least graduations. Each dial shall be equipped with a zero adjustment mechanism that can be operated from outside the dial case, and a maximum load indicator.

Section 4(b).—Add the following Note to Section 4(b): "NOTE.—Testing machines of any type with hand operated loading mechanisms, and hydraulic machines with only one loading rate, do not conform to these rate of loading requirements."

Revision of Standards,
Immediate Adoption

(requires nine-tenths affirmative vote)

Standard Method of Test for Weight per Cubic Foot, Yield, and Air Content (Gravimetric) of Concrete (C 138 – 44):

Section 6.—Add a new Paragraph (*d*) to read as follows, relettering the present paragraphs accordingly:

(*d*) *Relative Yield.*—Relative yield is the ratio of actual volume of concrete obtained to the volume as designed for the batch and shall be calculated as follows:

$$R_y = \frac{S}{V_d \times 27}$$

where:

R_y = relative yield,

S = volume of concrete produced per batch in cubic feet, and

V_d = volume of concrete which the batch was designed to produce in cubic yards.

NOTE.—A value for R_y greater than 1.00 indicates an excess of concrete being produced whereas a value less than this indicates the batch to be 'short' of its designed volume.

Standard Method of Test for Abrasion of Graded Coarse Aggregate by Use of the Deval Machine (D 289 – 55):

Section 5(a).—Add a Note to read: "NOTE.—For a given material, the grading used in the test may cause the percentage of wear obtained to vary to a marked extent. This should be observed in the preparation of specification requirements."

Section 5(c).—Delete this paragraph.

Standard Specifications for Ready-Mixed Concrete (C 94 – 62):

Section 3(d).—Delete from the second sentence the words "... for purposes other than entrainment of air ...", and make this sentence the first sentence and the present first sentence the second.

Section 7(b).—Delete the second sentence.

Adoption of Tentative Revision
As Standard

Standard Specifications for Ready-Mixed Concrete (C 94 – 62)

Incorporate the tentative revision of Section 9(*c*), issued June, 1961, as standard, including editorial revision of

Table I, as shown in the accompanying Table II.[5]

Adoption of Tentative as Standard Without Revision

Tentative Methods of Test for:

Static Young's Modulus of Elasticity and Poisson's Ratio in Compression of Cylindrical Concrete Specimens (**C 469 – 61 T**),
Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (**C 88 – 61 T**),
Sieve or Screen Analysis of Fine and Coarse Aggregates (**C 136 – 61 T**) (Jointly with Committee D-4), and
Ball Penetration in Fresh Portland Cement Concrete (**C 360 – 55 T**).

Tentative Specifications for:

Concrete Aggregates (**C 33 – 61 T**), and
Waterproof Paper for Curing Concrete (**C 171 – 60 T**).

Tentatives Continued Without Revision

Tentative Methods of Test for:

Compressive Strength of Concrete Using Portions of Beams Broken in Flexure (Modified Cube Method) (**C 116 – 60 T**),
Clay Lumps in Natural Aggregates (**C 142 – 55 T**),
Water Retention Efficiency of Liquid Membrane-Forming Compounds and Impermeable Sheet Materials for Curing Concrete (**C 156 – 55 T**),
Volume Change of Cement Mortar and Concrete (**C 157 – 60 T**),
Resistance of Concrete Specimens to Rapid Freezing in Air and Thawing in Water (**C 291 – 61 T**),
Resistance of Concrete Specimens to Slow Freezing in Air and Thawing in Water (**C 310 – 61 T**),
Sampling and Testing Fly Ash for Use as an Admixture in Portland Cement Concrete (**C 311 – 61 T**),
Potential Volume Change of Cement-Aggregate Combinations (**C 342 – 61 T**), and
Abrasion Resistance of Concrete (**C 418 – 58 T**),

Tentative Specifications for:

Lightweight Aggregates for Structural Concrete (**C 330 – 60 T**),
Lightweight Aggregates for Concrete Masonry Units (**C 331 – 59 T**), and
Single-Use Molds for Forming 6 by 12-in. Concrete Compression Test Cylinders (**C 470 – 61 T**).

Recommended Practice for:

Microscopical Determination of Air-Void Content Specific Surface, and Spacing Factor of the Air-Void System in Hardened Concrete (**C 457 – 60 T**).

Editorial Changes

Tentative Method of Test for Static Young's Modulus of Elasticity and Poisson's Ratio in Compression of Cylindrical Concrete Specimens (C 469 – 61 T):

Section 1.—Add a new last sentence as follows: "Chord modulus of elasticity and Poisson's ratio are defined in the Definition of Terms Relating to Methods of Mechanical Testing (ASTM Designation: E 6)."

Figures 1 and 2.—Add the word "Suitable" to the beginning of each caption.

Section 2(b).—Add a new last sentence as follows: "Procedures for calibrating strain-measuring devices are given in the Method of Verification and Classification of Extensometers (ASTM Designation: E 83)."

Tentative Method of Test for Splitting Tensile Strength of Molded Concrete Cylinders (C 496 – 62 T):

Section 2(b).—Change the third sentence to read: "It shall have a width of at least 2 in., and a thickness not less than the distance from the edge of the spherical or rectangular bearing block to the end of the cylinder."

Tentative Method for Making and Curing Concrete Compression and Flexure Test Specimens in the Field (C 31 – 62 T):

Section 5(c).—Change the last sentence to read: "After consolidation by any of the methods, the surface of the concrete shall be struck off without undue manipulation with a straightedge or trowel, the beams finished with a wood float, and all specimens shall be covered immediately with glass or metal plates, polyethylene film, or other covering to prevent evaporation."

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[5] Table II is not included in this report but appears in the revised specification; see 1963 Supplement to *Book of ASTM Standards*, Part 4.

Section 8(a).—After sentence ending ". . . stone plate (Note 7)." insert a new sentence as follows: "If a recess is machined into the metal plate, the thickness of the plate in the recessed area shall not be less than ½ in."

Section 9(c).—Immediately preceding the present last sentence, insert a new sentence as follows: "To meet these conditions the specimens shall be removed from the molds at the time of removing formwork."

Tentative Method for Making and Curing Concrete Compression and Flexure Test Specimens in the Laboratory (C 192 – 62 T):

Section 2(d).—In the second sentence, change "000" to read "7000."

This report has been submitted to letter ballot of the committee, which consists of 205 active members commanding 153 votes plus 10 honorary members having voting privileges; 186 members returned their ballots, of whom 185 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

BRYANT MATHER,
Chairman.

PAUL KLIEGER,
Secretary.

APPENDIX

THE INSPECTION OF CONCRETE TESTING LABORATORIES

REPORT OF SUBCOMMITTEE ON THE CEMENT AND CONCRETE REFERENCE LABORATORY

JOINT SUBCOMMITTEE OF COMMITTEES C-1 and C-9

The work of the Cement and Concrete Reference Laboratory (CCRL) in the field of concrete testing has been described briefly in two previous reports (1,2). Those accounts, however, do not cover certain accomplishments and various recent developments of interest in the concrete field prompts the preparation of this report.

RÉSUMÉ OF THE HISTORY OF THE CEMENT AND CONCRETE REFERENCE LABORATORY

The history of the Reference Laboratory begins in 1929. In April of that year, a Research Fellowship, known as the Cement Reference Laboratory, was established at the National Bureau of Standards under the sponsorship of ASTM Committee C-1 on Cement. The principal function of the newly created organization was to promote uniformity and improvement in the testing of cement through the field inspection of testing laboratories.

Several years later, when it became definite that the anticipated benefits were being obtained (3), the supporters of the Fellowship, each of whom was also much interested in concrete, began considering the feasibility of extending the inspection work to cover concrete testing. The onset of World War II postponed definite action along these lines until 1948 when the first step toward broadening the scope of the program was taken by providing for the verification of the large-capacity compression machines used for the testing of concrete in the cement testing laboratories which were then receiving inspections. The results were encouraging, and subsequently coverage was expanded to include most of the methods and equipment used in determining the compressive strength of concrete.

On March 1, 1958 (4), the inspection service thus evolved was made available to any interested concrete testing laboratory within the prescribed areas of operation, and shortly thereafter Committee C-1 invited Committee C-9 to become a joint sponsor of the Fellowship. The invitation was accepted, and on July 1, 1960 (5), the project was renamed "The Cement and Concrete Reference Laboratory" in recognition of the new arrangement.

Prior to the redesignation, the operation of the laboratory had been cooperatively administered by the Inorganic Building Materials Section of the Building Research Division of the National Bureau of Standards and the Subcommittee on the Cement Reference Laboratory of Committee C-1. Under the new ASTM sponsorship plan, a joint C-1, C-9 Subcommittee on the Cement and Concrete Reference Laboratory, consisting of seven representatives of each of the parent committees, was created to work with the National Bureau of Standards. The new plan perpetuated the previous arrangement in which representatives of other Federal Agencies and several of the product manufacturers and national trade associations, who furnish both financial and technical assistance, participate in the direction of the Fellowship through membership on the supervising ASTM Subcommittee.

SCOPE

At the request of Committee C-9 a scope was written for its new seven-man Subcommittee on the CCRL, as follows:

> "To promote uniformity and improvement in the testing of cements and concretes through the field inspection of testing laboratories, the study of special problems evolving from such inspections, instruction in methods of testing, sponsorship of comparative test programs, and contributions to the work of technical committees."

This statement outlines the purpose and function of the Cement and Concrete Reference Laboratory as well as describing the responsibilities of the Joint C-1, C-9 Subcommittee.

THE INSPECTION SERVICES

The inspection of testing laboratories is considered to be the most important function of the CCRL. The services are advisory in nature and are made available only to those laboratories who are prepared to make the standard ASTM tests. Utilization is on a voluntary basis and there is no direct regulatory action involved.

The program for concrete laboratories presently provides for examination of the apparatus used in measuring the slump of fresh concrete, the compressive strength of hardened concrete, and the mortar making properties of fine aggregates. Instruction is also given in the related methods of test. The more important matters dealt with can be described as follows:

1. The salient features of the laboratory work room,
2. The physical condition and dimensions of apparatus used in the slump test,
3. The suitability of cylinder molds,
4. The adequacy of moist storage facilities,
5. The conformance of capping equipment with specification requirements,
6. The conformance of capping techniques with prescribed procedures and standard practices,
7. The proper handling of cylinders between capping and testing,
8. The accuracy of indication of the compression testing machine and the condition and size of its bearing blocks, and
9. The acceptability of the apparatus used in measuring the mortar making properties of fine aggregates.

The inspection of a separate concrete testing laboratory takes approximately 8 hr. A part, if not all, of one hour is used in the unloading and reloading of the 500 lb. of circular proving rings and accessory equipment, gages and micrometers, thermometers, psychrometers, small tools and reference literature employed in the inspection work. At least 6 hr are needed for carrying out the operations described in the preceding paragraph. The concluding hour is devoted to a detailed oral report which the CCRL Inspector presents to interested members of the laboratory staff prior to his departure.

No rating or certification is given to the laboratory on the basis of the inspection. However, as promptly as possible, a detailed confirmatory report, which contains an informal document that may be displayed as an evidence of the inspection, is sent to the individual who requested the visit. Much careful effort is devoted to the preparation of inspection reports because they are often the basis upon which laboratories initiate repair or replacement of faulty equipment or upon which decisions are reached about their capability to perform certain types of testing work.

Inspection observations, and consequently confirmatory reports, are considered to be confidential in nature. Accordingly, the communication of findings is largely restricted to persons associated with the participating laboratory. However, the CCRL will distribute copies of an inspection report to other interested parties when authorized to do so by the laboratory concerned. For more than 25 yr, the Bureau of Public Roads has used this procedure to obtain information about testing laboratories engaged in work related to Federal Aid Projects, and several other organizations are now considering the feasibility of taking similar action where the laboratories in which they are interested are concerned.

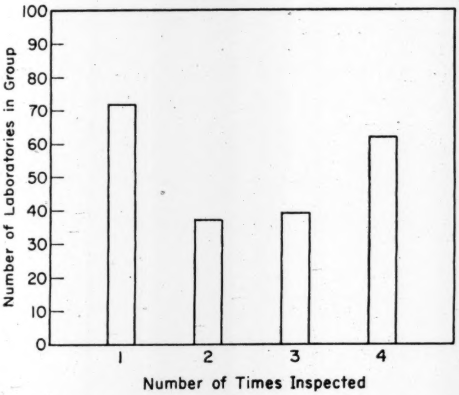

FIG. 1.—Inspection Record of 210 Concrete Laboratories.

TABLE I.—CLASSIFICATION AND NUMBER OF CONCRETE LABORATORIES INSPECTED.

Laboratory Classification	Tour 10	Tour 11	Tour 12	Tour 13
Cement Producer	22	20	19	18
Commercial Testing	38	24	45	54
Highway Department	43	37	41	47
University	8	3	2	5
Municipal	4	1	2	1
Federal	9	8	11	12
Miscellaneous	7	5	11	14
Total Inspections	131	98	131	151
Number of New Participants	...	17	32	30
Approximate Tour Period	1950 to 1954	1955 to 1957	1957 to 1960	1960 to 1962
Number of Cement Laboratories Inspected in Tour	261	253	266	291

TABLE II.—CONFORMANCE OF APPARATUS EXAMINED WITH SPECIFICATION REQUIREMENTS IN LABORATORIES PARTICIPATING IN AT LEAST FOUR INSPECTIONS.

	Tour 10—Conforming, per cent	Tour 11—Conforming, per cent	Tour 12—Conforming, per cent	Tour 13—Conforming, per cent
Moist Storage Facilities	79	68	63[a]	61
Capping Plates	40	55	86	90
Watertightness of Cylinder Molds:				
Single-use type	...	...	77	71
Reusable type	...	...	28	40
Compression Machines:				
Accuracy, per cent:				
within ±1	56	59	64	55
within ±2	81	71	80	76
within ±3	90	86	91	91
within ±10	99	99	99	100
within ±20	99	100	100	100
Bearing Blocks:				
size of spherical (Method C 39)	86	86	97	97
planeness	63	69	79	82
concentric circles (Method C 39)	72	80	85	91

[a] Major change in requirements.

NUMBER AND INTEREST OF INSPECTED LABORATORIES

The field program is designed to provide for the re-inspection of regularly participating laboratories at intervals of from 24 to 30 months, and it is customary to refer to each complete circuit as an "inspection tour." The Ninth Inspection Tour was nearing completion when the concrete work was inaugurated in 1948, and therefore summations of information relating to this activity are customarily started with the Tenth Tour.

As of June 1, 1962, a total of 511 inspections had been performed in the 210 concrete laboratories that had been visited since the start of the Tenth Tour on July 10, 1950. The bar-graph in Fig. 1 shows the number of times each has been inspected.

TABLE III.—CONFORMANCE OF APPARATUS EXAMINED WITH SPECIFICATION REQUIREMENTS IN ALL OF THE LABORATORIES INSPECTED.

	Tour 10—Conforming, per cent	Tour 11—Conforming, per cent	Tour 12—Conforming, per cent	Tour 13—Conforming, per cent
Moist Storage Facilities	58	63	54[a]	53
Capping Plates	48	53	72	69
Watertightness of Cylinder Molds:				
Single-use type	...	...	73	69
Reusable type	...	...	22	25
Compression Machines:				
Accuracy, per cent:				
within ±1	50	56	53	49
within ±2	75	75	74	73
within ±3	84	86	83	88
within ±10	98	98	98	97
within ±20	98	100	99	99
Bearing Blocks:				
size of spherical (Method C 39)	85	88	98	92
planeness	69	56	62	74
concentric circles (Method C 39)	85	80	84	90

[a] Major change in requirements.

The number of laboratories participating in each of the four tours, Tenth to Thirteenth inclusive, and a general classification according to the interest represented are shown in Table I. The number of cement laboratories visited during each tour has also been tabulated in order to show the magnitude of the inspection work when both cement and concrete laboratories are counted.

BENEFITS OF THE INSPECTION WORK

Because of the restrictions on participation and the liberality of the requirements for apparatus at the time the program was implemented, it is difficult to demonstrate all of the probable benefits of the inspection work in the concrete field. However, a general concept of its effect can be obtained from a selective study of inspection findings concerning apparatus.

The data in Table II is derived by limiting the study to the laboratories that have been inspected in each of the four tours under discussion. The importance of the improvements in uniformity here reported is demonstrated by the estimation that collectively these laboratories test more than 500,000 concrete cylinders per year.

The data in Table III is derived by extending the study to include findings in all of the laboratories inspected during each tour. In general, the differences between corresponding percentages in Tables II and III support the hypothesis that the inspection service is most effective when it is carried out and used on a regular basis.

THE STUDY OF SPECIAL PROBLEMS

The study of questions evolving from the inspection work has been stated as a second important function of the CCRL. In practice, this part of the program is usually carried forward as a coordinated laboratory-field operation with the principal aims being the development of information upon which revisions in methods of test or changes in inspection procedures might be based.

The time that can be devoted to this activity is limited because primary emphasis is placed on satisfactory accomplishment of the field inspection work. The necessity for giving equal attention to problems relating to the testing of cement further limits the time that can be alloted to concrete testing problems. It is believed, however, that the work has been of value in spite of these limitations, and the following brief discussions of three proceedings are presented to support this conclusion.

Compression Machine Bearing Blocks.—A wide variation in the dimensions of the circular and rectangular blocks used in the testing of 6 by 12-in. concrete cylinders was discovered during the early inspections in concrete testing laboratories. The findings were tabulated and described in a special report on the inspection work that was presented to Committee C-9 in 1953. The data proved to be of considerable interest and reference was made to it on several subsequent occasions during the discussions which preceded the adoption of limiting dimensions and certain other new requirements for bearing blocks in 1956. The resulting improvement in uniformity is shown in Table II.

Sulfur Capping Materials.—The fabrication of cylinder caps from free sulfur and cracking or shrinkage in sulfur-mortar caps were observed on many occasions during the inspections performed between 1950 and 1955. These observations suggested that special techniques for demonstrating the suitability or unsuitability of sulfur capping materials were needed, and experiments along these lines were initiated in 1956. As a result, the CCRL is now prepared to evaluate the quality of each material encountered by means of its compressive strength and, when appropriate, to offer suggestions for the improvement of inferior sulfur-mortar caps based on a determination of their sulfur content, the gradation of the granular materials, and the melting temperature used. Advantages accruing from this improvement in inspection procedures are the reductions in the use of uncombined sulfur and miscellaneous mixtures that have occurred since the Tenth Tour, and the recommendations which the CCRL has made for changes in the requirements for capping equipment and materials set forth in ASTM Methods C 31 and C 192.

TABLE IV.—ABSORPTION AND EXPANSION DATA FOR PAIRS OF PAPER MOLDS.

Source	Approximately 4-hr Elongation, in.		Weight Gain in 4 hr, g		Water-tightness[c]
	Mold No. 1	Mold No. 2	Mold No. 1	Mold No. 2	
PAPER MOLDS WITH PAPER BOTTOMS					
1.....	0.05	...	[b]	...	U
1.....	0.00	0.00	[b]	[b]	U
2.....	0.02	0.01	25	18	U
2.....	[a]	[a]	[a]	[a]	U
3.....	0.03	0.04	3	4	S
3.....	0.05	0.06	26	20	U
4.....	0.08	0.03	[a]	[a]	U
5.....	[b]	[b]	4	6	S
6.....	[a]	[a]	13	14	S
6.....	0.00	0.00	6	6	S
6.....	0.06	0.06	28	24	U
PAPER MOLDS WITH METAL BOTTOMS					
5.....	[a]	[a]	125	123	S
5.....	0.00	0.00	14	17	S
6.....	0.03	0.00	[b]	[b]	S
6.....	0.00	0.00	[b]	[b]	S
6.....	0.00	0.00	[b]	[b]	S
6.....	0.03	0.03	[b]	[b]	S
6.....	0.12	0.14	3	5	S
6.....	0.00	0.00	[b]	[b]	S
6.....	0.00	0.02	7	10	S
6.....	0.02	0.09	4	2	S
6.....	0.00	0.00	3	2	S
6.....	0.02	0.02	24	20	S
6.....	(−)0.01	0.01	7	5	S
7.....	0.00	0.00	[b]	[b]	S
7.....	0.01	0.00	17	5	S
7.....	0.15	0.23	47	103	U
9.....	[a]	[a]	[a]	[a]	U

[a] Observation could not be made.

[b] No significant change.

[c] S = Satisfactory.
U = Unsatisfactory.

Cylinder Molds.—A number of seemingly important differences in the physical properties of cylinder molds were noted during the Tenth Inspection Tour. It was subsequently concluded that a performance test was needed, and after considerable experimentation a test procedure similar to that which is now being used as the standard test for single-use molds was incorporated in the inspection procedures in 1957. As a consequence, the CCRL was in a position to furnish absorption and expansion data for paper molds, such as that shown in Table IV, to the task group appointed in June, 1958, to write the recently adopted tentative specifications for such molds.

INSTRUCTIONS IN METHODS OF TESTING

Instruction in methods of testing is normally accomplished as part of the inspection work. It is important to note, however, that the CCRL is occasionally afforded opportunities to participate in the special courses of instruction for concrete technicians that are periodically presented in various sections of the United States. In 1953, and again in 1961, the Manager of the Reference Laboratory lectured at the annual Quality Concrete Conference held at the University of North Carolina. The 1961 paper was subsequently published in the July, 1962, edition of *Modern Concrete.* In November, 1959, a paper entitled "Requirements for a Concrete Testing Laboratory; and the Function of the Cement and Concrete Reference Laboratory" was presented at the Fourteenth Annual Short Course of Instruction on Concrete and Concrete Aggregates held jointly by the National Ready Mixed Concrete Assn and the National Sand and Gravel Assn at the University of Maryland. This paper was also presented in January, 1960, at the first Short Course on Concrete and Concrete Aggregates held at Northeastern University, Boston, Mass.

SPONSORSHIP OF COMPARATIVE TEST PROGRAMS

The sponsorship of comparative test programs has been limited to the work among cement testing laboratories, and therefore is not an item of importance where this discussion is concerned.

PARTICIPATION IN THE WORK OF TECHNICAL COMMITTEES

Participation in the work of technical committees has been made one of the major functions of the Cement and Concrete Reference Laboratory. The assignments of possible importance where the testing of concrete is concerned are as follows:

1. Representation of Committee C-1 on Committee C-9; and on the E-1 Subcommittees on Calibration of Mechanical Testing Machines and Apparatus, Effect of Speed in Mechanical Testing, Conditioning and Weathering, and Metalware Laboratory Apparatus.
2. Membership on C-9 Subcommittees III-a on Methods of Testing Concrete for Strength, III-p on Manual of Concrete Testing, and III-r on Organic Materials for Bonding, Patching, and Sealing Concrete.
3. Membership on American Concrete Inst Committee 116 on Nomenclature.

The inspection of concrete testing laboratories is an expanding activity of the Cement and Concrete Reference Laboratory. It is believed that the anticipated benefits are being obtained and that the expansion should be continued.

Respectfully submitted on behalf of the joint C-1, C-9 Subcommittee on the Cement and Concrete Reference Laboratory,

CLAYTON L. DAVIS (C-1),
Chairman.

Subcommittee Members

HAROLD ALLEN (C-9)
A. A. BATES (C-1)
R. L. BLAINE (C-1)
H. K. COOK (C-9)
J. G. DEMPSEY (C-9)
J. L. GILLILAND (C-1)
H. F. HEDDERICH (C-9)
R. R. LITEHISER (C-1)
W. H. PRICE (C-9)
M. R. SMITH (C-1)
STANTON WALKER (C-9)
J. H. WALKER (C-1)
E. A. WHITEHURST (C-9)

REFERENCES

(1) "History of the Cement Reference Laboratory 1929–1955," Report of Committee C-1, Appendix I, *Proceedings,* Am. Soc. Testing Mats., Vol. 57, p. 232 (1957).

(2) J. R. Dise, "The Cement Reference Laboratory (1929–1959)," Report of Committee C-1, Appendix II, *Proceedings,* Am. Soc. Testing Mats., Vol. 59, p. 369 (1959).

(3) Report of Committee C-1 for 1935, *Proceedings,* Am. Soc. Testing Mats., Vol. 35, Part I, p. 238 (1935).

(4) "Services of Cement Reference Laboratory Extended to Concrete," ASTM BULLETIN, No. 228, Feb., 1958, p. 16.

(5) Report of Committee C-1 for 1960, *Proceedings,* Am. Soc. Testing Mats., Vol. 60, p. 252 (1960).

REPORT OF COMMITTEE C-11
ON
GYPSUM*

Committee C-11 on Gypsum held two meetings during the year: in New York, N. Y. on June 28 and 29, 1962, and in Montreal, Canada on Feb. 4 and 5, 1963.

The committee consists of 39 members, of whom 34 are voting members; 15 are classified as producers, 10 as consumers, 9 as general interest members, 1 as a consulting member, and 4 alternates.

Committee C-11 is in the process of conducting two test programs in private laboratories. One program concerns the testing of various perlites for use with gypsum perlite plaster to provide further information on perlite aggregates such as compressive strength, shrinkage, and the performance characteristics of gypsum perlite plaster that may be due to the use of perlite aggregates.

The other program concerns the verification of an adhesive test method which is currently being checked to determine reproducibility of the method. The results of this work should be concluded within the year and will lead to a new specification.

The test program investigating the conical plunger method of determining the normal consistency of plaster has been completed. As a result, Subcommittee I is currently recommending to Committee C-11 that changes be made in Method C 472 whereby the conical plunger method for determining the normal consistency of plaster will be incorporated.

Recommendations for Society Action at Annual Meeting

Committee C-11 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] All of the existing tentatives and standards referred to appear in their present form in the 1961 Book of ASTM Standards, Part 4.

New Tentative

Tentative Performance Specification for Nails for the Application of Gypsum Wallboard, as appended hereto.[2]

Adoption of Tentatives as Standard Without Revision

Tentative Specifications for:

Precast Reinforced Gypsum Slabs (**C 377 - 58 T**),
Annular Ringed Nails for Gypsum Wallboard (**C 380 - 61 T**), and
Gypsum Backing Board (**C 442 - 61 T**).

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[2] The new tentative appears in the 1963 Supplement to *Book of ASTM Standards*, Part 4.

Revision of Standard, Immediate Adoption

(requires nine-tenths affirmative vote)

Standard Specifications for Gypsum Plasters (**C 28 – 60**):
Section 3.—Change to read as follows:

3. Gypsum ready-mixed plaster is a calcined gypsum plaster, mixed at the mill with a mineral aggregate, designed to function as a base to receive various finish coats. It may contain other materials to control setting time and the desirable working properties.

Tentatives Continued Without Revision

Tentative Methods of Testing Joint Treatment Materials for Gypsum Wallboard Construction (**C 474 – 61 T**), and

Tentative Specifications for Joint Treatment Materials for Gypsum Wallboard Construction (**C 475 – 61 T**).

This report has been submitted to letter ballot of the committee, which consists of 34 voting members; 29 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

G. W. JOSEPHSON,
Chairman.

H. OMSON,
Secretary.

REPORT OF COMMITTEE C-12
ON
MORTARS FOR UNIT MASONRY*

Committee C-12 on Mortars for Unit Masonry held two meetings during the year: on June 27, 1962, in New York, N. Y., and on Feb. 19, 1963, in Washington, D. C.

The committee consists of 51 voting members, of whom 23 are classified as producers, 12 as consumers, and 16 as general interest members. In addition, there are six consulting members.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee C-12 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] The existing tentatives referred to in this report appear in their present form in the 1961 Book of ASTM Standards, Part 4.

Adoption of Tentative as Standard Without Revision

Tentative Specification for Mortar and Grout for Reinforced Masonry (**C 476 – 61 T**).

Tentative Continued Without Revision

Tentative Specification for Mortar for Unit Masonry (**C 270 – 61 T**).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Research and Methods of Test (C. U. Pierson, Jr., chairman) has undertaken cooperative research on mortar in ten laboratories on: (1) use of the Emley plasticimeter in the water retention test, (2) efflorescence testing, (3) compressive strength of mortar at field consistency, and (4) a new consistency test for field use.

Subcommittee III on Specifications (J. Lee, chairman) is preparing extensive revisions of the Specifications for Mortar for Unit Masonry (C 270).

Subcommittee IV on Aggregates for Mortar (D. L. Bloem, chairman) is studying the physical and economical aspects of aggregate grading as related to mortar.

Subcommittee V on Organic-Modified Mortars (P. M. Woodworth, chairman) a new subcommittee, is preparing specifications and methods of test for mortars for masonry that are modified with organic polymers.

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

This report has been submitted to letter ballot of the committee, which consists of 51 members; 38 members returned their ballots, of whom 37 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

H. C. PLUMMER,
Chairman.

H. TOENNIES,
Secretary.

REPORT OF COMMITTEE C-13
ON
CONCRETE PIPE*

Committee C-13 on Concrete Pipe held one meeting during the year: in Chicago, Ill. on Oct. 19, 1962.

The committee consists of 50 voting members, of whom 23 are classified as producers, 13 as consumers, and 14 as general interest members.

The Special Committee on Rubber Gaskets has been renamed Subcommittee VIII on Rubber Gaskets. The Special Committee on Separation of Tests has been renamed Subcommittee IX on Test Procedures. A new Subcommittee X on Correlation and Clarification has been appointed. Its membership consists of the chairmen of all other Subcommittees, and its chairman will always be the chairman of Subcommittee IX on Test Procedures.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee C-13 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] All of the existing standards and tentatives referred to appear in their present form in the 1961 Book of ASTM Standards, Part 5, or the 1962 Supplement thereto.

New Tentatives

as appended hereto.[2]

Tentative Specifications for:

Nonreinforced Concrete Irrigation Pipe With Rubber-Type Gasket Joints,

Reinforced Concrete, Elliptical Culvert, Storm Drain, and Sewer Pipe, and

Reinforced Concrete, Arch Culvert, Storm Drain, and Sewer Pipe.

Revision of Tentatives

Tentative Specifications for Reinforced Concrete Culvert, Storm Drain, and Sewer Pipe (C 76 - 62 T):

Section 6.—Delete the words "structural or" and add in the same line the word "steel" between the words "grade" and "conforming."

Section 13.—After the first sentence add a new sentence to read: "In addition, where lapped cages of welded wire fabric are used without welding, the lap shall contain a longitudinal wire." In the present second sentence change "75 per cent" to read "50 per cent," and add after the last word "steel", the words: "and there shall be a minimum lap of 2 in."

Tables II, III, IV, and V.—Change the steel areas for 12- through 24-in.

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[2] The new tentatives appear in the 1963 Supplement to *Book of ASTM Standards*, Part 5.

diameter pipe for those portions shown in the accompanying Table I, including a new footnote for Tables II and III which is designated by an asterisk. The delete the words "two layers" and substitute the words "a layer." Also, delete the words "in each layer" in the same sentence.

TABLE I.—DESIGN REQUIREMENTS FOR REINFORCED CONCRETE PIPE.
(Revisions in Tables II, III, IV, and V of Specification C 76)

Diameter	Wall A			Wall B			Wall C			
	Wall	Circular Reinforcement	Elliptical Reinforcement	Wall	Circular Reinforcement	Elliptical Reinforcement	Wall	Circular Reinforcement		Elliptical Reinforcement
								Inner Cage	Outer Cage	
CLASS II (Table II)										
12	1¾	0.07*	...	2	0.07*	...	...	...	...	...
15	1⅞	0.07*	...	2¼	0.07*	...	...	...	...	...
18	2	0.07*	0.07*	2½	0.07*	0.07*	...	...	...	...
21	2¼	0.12	0.10	2¾	0.07*	0.07*	...	...	...	...
24	2½	0.13	0.11	3	0.07*	0.07*	...	...	...	...
CLASS III (Table III)										
12	1¾	0.07*	...	2	0.07*	...	...	...	...	...
15	1⅞	0.07*	...	2¼	0.07*	...	...	...	...	...
18	2	0.07*	0.07*	2½	0.07*	0.07*	...	...	...	...
21	2¼	0.14	0.11	2¾	0.07*	0.07*	...	...	...	...
24	2½	0.17	0.14	3	0.07*	0.07*	...	...	...	...
CLASS IV (Table IV)										
12	1¾	0.15	...	2	0.07	...	...	...	...	...
15	1⅞	0.16	...	2¼	0.10	...	...	...	...	...
18	2	0.17	0.15	2½	0.14	0.11	...	...	...	...
21	2¼	0.23	0.21	2¾	0.20	0.17	...	...	...	...
24	2½	0.29	0.27	3	0.27	0.23	3¾	0.07	0.07	0.08
CLASS V (Table V)										
12	b	...	...	2	0.10	...	...	...	...	...
15	b	...	...	2¼	0.14	...	...	...	...	...
18	b	...	...	2½	0.19	0.16	...	...	...	...
21	b	...	...	2¾	0.24	0.21	...	...	...	...
24	b	...	...	3	0.20	0.24	3¾	0.12	0.09	0.13

* For these classes and sizes, the minimum practical steel reinforcement is specified since equivalent strengths are available in nonreinforced pipe in the Specification for Concrete Sewer Pipe (ASTM Designation: C 14).

Footnote *b* referenced for class V refers to the existing Footnote *b*.

Tentative Specifications for Precast Reinforced Concrete Manhole Risers and Tops (C 478 – 61 T):

Section 9(c).—In the second sentence

Section 11(b).—Delete the first sentence and substitute the following two sentences:

> In flat slab tops the layer of reinforcement shall be placed near the bottom surface so that the nominal protective cover over the reinforcement shall be one inch. Flat slab tops manu-

factured without a joint or other indication of the top or bottom of the slab shall be manufactured with two layers of steel reinforcement, one located near the bottom surface and one near the top surface so that the nominal protective cover over each layer is 1 in.

Tentative Specifications for Joints For Circular Concrete Sewer and Culvert Pipe, Using Flexible, Watertight Rubber-Type Gaskets (C 443 - 62 T):

Section 1.—Delete Paragraph (*b*)

Section 4.—Designate present paragraph as (*a*). In the tabular material add the word "nominal" after "Shore durometer hardness."

Add a new Paragraph (*b*) to read as follows:

(*b*) If a splice is used in the manufacture of the gasket, the strength shall be such that the gasket shall withstand 100 per cent stretch over that part of the gasket including the splice with no visible separation.

Section 5.—In Paragraph (*b*) line 9, change "8 deg" to read "3½ deg." In line 11 change "10 deg" to read "8 deg." In line 7 and 8 delete the words "should be as small as practicable and."

In Paragraph (*d*) line 2, after the word "design" add "when tested under Section 8."

Tentative Methods of Test for Determining Physical Properties of Concrete Pipe or Tile (C 497 - 62 T):

Section 2(a).—Delete the last sentence.

Section 3.—Delete the first sentence.

Section 5(e).—Delete the word "appreciable" from the end of the first sentence and add after the word "deflection" the words "greater than provided in the Note in Table I."

Section 6(c).—Change the word "or" in the last sentence to read "and."

Section 13.—Change to read as follows under the heading "Specimens":

13. Absorption test specimens may be obtained from acceptable strength test specimens if and after that test has been made. Each specimen shall have a minimum weight of 100 g, shall be free from visible cracks, and shall represent the full wall thickness of the pipe or tile.

Section 15.—In line 3 delete the words "of concrete drain tile."

Section 16.—Delete this section and renumber subsequent sections accordingly.

Sections 19 to 22.—Delete these sections on the Freezing-and-Thawing Test.

Tentative Specifications for Cast-In-Place Nonreinforced Concrete Irrigation Closed Conduit (C 477 - 61 T):

Title.—Change to read "Cast-in-place nonreinforced concrete agricultural closed conduit."

Section 1.—Change to read as follows:

1. These specifications cover cast-in-place nonreinforced concrete agricultural conduit suitable for the conveyance and distribution of irrigation and agricultural drainage water under low hydrostatic pressures (not over 15 ft maximum working head above the center line of the conduit), as installed in stable soils where the trench bottom and sides are used as the outside form for approximately the bottom half of the conduit. Irrigation systems shall be designed with vents and open stands so as to prevent damage from air action and water hammer. The specifications do not provide for additional loads that may be encountered under public roads, high fills, or structures.

Section 3.—Change the last sentence to read: "Any defective portion of the pipe shall be replaced or repaired to meet these specifications at the expense of the contractor." In the first sentence after the word "specified," add the words "if and when required."

Section 4.—In Paragraph (*a*) change "210" to read "180."

In Paragraph (*c*), beginning with line 13, change to read: ". . . side of the normal trench wall shall be excavated and the space refilled with selected cohesive soils compacted by acceptable methods to a density that will provide stability and permit excavation without sloughing."

In Paragraph (*d*) delete the words "acceptable rubber gasket joints and."

In Paragraph (*e*) change "undistributed" to "undisturbed."

Section 5.—Change to read as follows:

5. Departure from and return to established grade shall not exceed 1 in. per 10 lineal ft, and the maximum departure shall be limited to 1½ in. Departure from and return to established alignment shall not exceed 2 in. per 10 lineal ft and maximum departure shall be limited to 4 in.

Section 7.—Following the second sentence, insert: "Other admixtures or blends may be used with the approval of the purchaser." Change the present fourth sentence to begin: "The slump, determined for each 25 cu yd of concrete, and made in accordance with . . ." Delete last sentence.

Section 8.—Change to read as follows:

8. The ASTM designation and type of cement to be used shall be specified by the purchaser when calling for bids. In case this is not done, bidders shall ask the purchaser to so specify in advance of bid date. If the purchaser does not then comply, the successful bidder shall determine the ASTM designation and type of cement to be used.

Table I.—Position Table I before Section 12, and add the following sizes: "54-in. I.D. with 5-in. wall; 60-in. I.D. with 5½-in. wall."

Section 14.—Delete the first two sentences and substitute: "Offsets at form laps, both transverse and longitudinal, shall not exceed the thickness of the form plus ¼ in."

Section 15.—Change to read as follows:

15. Compressive strength of 28-day concrete as determined from standard 6- by 12-in. cylinders, as specified in Section 26, made from concrete taken from each 25 cu yd, or from cores where required, shall average at least 3000 psi for each day's placement, but no cylinder shall test less than 2400 psi. If cylinder strength for any day's placement is unsatisfactory, the contractor shall take cores as specified in Section 27.

Section 16.—Delete this section, renumbering subsequent sections.

Section 17.—Change heading to read "Hydrostatic Test."

Section 18.—Change to read: "The pipe shall be constructed in one placement."

Section 19.—Delete this section.

Section 20.—Delete the portion of the second sentence following the word "vertical."

Section 21.—Change to read: "21. Concrete shall not be placed when temperature of the concrete exceeds 90 F or is less than 50 F. The soil adjacent to the trench shall be at a temperature above freezing."

Section 22.—Change the first sentence to read: "The contractor shall be responsible for seeing that the concrete is properly cured."

Section 22.—In Item (*1*) transfer the words "which shall be kept moist" to the end of the sentence.

In Item (*2*) add the following sentence at the end: "Polyethylene film, at least 0.0015 in. thick, or other approved waterproof mats covering the exposed concrete surface may be used in lieu of a spray curing compound."

In the first paragraph of Item (*3*) delete "to a minimum depth of 6 in." In the first sentence of the second paragraph, delete "immediately." Following the words "after placement" insert "as soon as possible without damage to the concrete." Revise the second part of the paragraph to read as follows:

In the case of the method described in Paragraph (*1*), the backfill shall be placed immediately behind the removal of moisture-retaining material. The initial backfill for all methods shall be kept moist until at least 12 in. more of backfill is placed. Final backfill shall not be started until the pipe has developed sufficient strength to support the loads to be imposed. All openings into the pipe shall be kept closed at all times during construction, except when and where work is in progress, to keep air drafts from drying out the concrete.

Section 24.—In line 2 change "shall" to read "may."

Section 25.—Change the first sentence to read: "As soon as practical after placement and after the inside forms have been removed, rock pockets, cracks, and indentations shall be repaired."

Section 27.—Change to read as follows:

> 27. When required, cores shall be obtained from the pipe not sooner than 5 days after placement, and tested in accordance with the Methods of Securing, Preparing, and Testing Specimens from Hardened Concrete for Compressive and Flexural Strengths (ASTM Designation: C 42). Tests of all cores shall be for length (wall thickness) in accordance with the Method of Measuring Length of Drilled Concrete Cores (ASTM Designation: C 174), and for compressive strength in accordance with Method C 39. The diameter of the cores shall provide a length divided by diameter ratio of not less than one. The contractor, when required to do so by the provisions of Section 15, shall obtain 4 cores for each day's placement, at locations selected by the purchaser. The contractor shall patch all core holes in such a manner that the patch will be permanent, will not leak, and will have a smooth interior surface flush with the pipe intrados.

TABLE II.—DESIGN AND PHYSICAL TEST REQUIREMENTS.
(Revised Table I for Specification C 118)

Internal Diameter, in.	Wall Thickness, in.	Minimum Internal Hydrostatic Pressure, psi	Minimum Three-Edge Bearing Load, lb. per lineal ft
4	¾	50	1200
5	¾	50	1250
6	⅞	50	1300
8	1	50	1350
10	1⅛	50	1400
12	1¼	45	1500
14	1⅜	45	1600
15	1½	45	1650
16	1½	45	1700
18	1¾	45	1800
20	2	40	1850
21	2⅛	40	1900
24	2¼	40	2000

Section 28.—Change the first sentence to read: "The thickness of the pipe shall be measured every 50 lineal ft, with individual measurements staggered at points designated by the purchaser."

Section 29.—Change the heading to read "Hydrostatic Test." In the first sentence after the words "for 2 weeks," insert the phrase "or earlier if mutually agreed upon." In the fourth sentence, insert the word "static" between the words "maximum" and "design."

Over-all Revision.—Delete all reference to the word "pipe" in the body of the specification and substitute the words "closed conduit."

Revision of Standards
Immediate Adoption

(requires nine-tenths affirmative vote)

Standard Specifications for Concrete Sewer Pipe (C 14 – 59):

New Section.—Add a new section on curing in the appropriate place to read the same as Section 17 in Specifications C 76 with the following exception: In the first paragraph delete the word "compressive." Insert the word "requirement" in the last sentence after the word "strength."

Standard Specifications for Concrete Pipe for Irrigation or Drainage (C 118 – 60):

Section 8.—Change to read as follows under the heading "Design":

> 8. Design requirements shall be in accordance with Table I. Wall thickness used may be more than but not less than the values shown, except as affected by the tolerance herein specified.

Table I.—Change to read as shown in the accompanying Table II.

Section 11.—Delete the words "Crushing Strength" from the heading.

Section 13.—Change the second sentence to read: "Should any of the initial test specimens fail to meet the test requirements, the manufacturer will be allowed a retest on two additional specimens for each specimen that failed, and pipe from the tested lot shall be acceptable only when all of these retest specimens meet the test requirements."

Section 15.—Change the heading to read "Permissible Variations in Dimensions." Designate present paragraph as (*a*) and add a new Paragraph (*b*) to read: "(*b*) The minimum wall thickness of the pipe barrel shall not be more than 5 per cent, or $\frac{1}{16}$ in., whichever is greater, less than the wall thickness specified in Table I."

TABLE III.—PHYSICAL TEST REQUIREMENTS FOR STANDARD QUALITY CONCRETE DRAIN TILE.

(Revised Table I for Specification C 412)

Nominal Inside Diameter, in.	Standard-Quality Concrete Drain Tile				
	Three-Edge Bearing Crushing Strength^{a}			Absorption, boiled 5 hr	
	Minimum Average, lb per lin ft		Minimum for Individual Tile, lb per lin ft	Maximum Average, per cent	Maximum for Individual Tile, per cent
	A	B	A and B	B	B
4	900	800	700	10	11
5	900	800	700	10	11
6	900	800	700	10	11
8	900	800	700	10	11
10	900	800	700	10	11
12^{b}	900	800	700	10	11

Section 16.—Change to read: "Pipe shall be substantially free of fractures large or deep cracks, and surface roughness."

Section 17.—Delete the words "and have been properly cured for at least 7 days."

Section 19.—Change to read as follows:

19. Individual pipe units shall be subject to rejection on account of failure to conform to any of the specification requirements or on account of any of the following:

(*a*) Fractures or cracks passing through the shell shall be cause for rejection, except for an end crack that does not exceed the depth of the joint, or a fracture that at its deepest point does not exceed the depth of the joint nor extend more than 10 per cent around the circumference of the joint. (No change in Paragraphs (*b*) and (*c*).)

Section 20.—Delete the words "Crushing Strength" from the centered heading.

Section 21(a).—Change to read as follows:

21. (*a*) The load shall be applied parallel to and along a vertical plane passing through the axis of the pipe, as shown in Fig. 1. The lower bearings shall consist of two wooden strips not less than 1 in. nor more than 1½ in. in height and not more than 2 in. wide at the base, having their interior top corners rounded to a radius of approximately ½ in. The strips shall be straight and shall be securely fastened to a rigid uniformly supported wooden block at least 6 in. wide. The interior vertical sides of the strips shall be parallel and spaced a distance apart of not more than 1 in. per ft of pipe diameter, but in no case less than 1 in. The upper bearing shall be a rigid wooden block, free of knots, and straight and true from end to end. The load shall be applied to this block through a metal beam of such dimensions that it will transmit the full load without appreciable deflection. Sand or other loose fine material may be spread along the length of the crown of the pipe to equalize the upper bearing. Pipe having exterior surface irregularities may have the depressions filled with plaster of Paris to equalize the bearing surfaces. The upper and lower bearings shall extend the full length of the pipe exclusive of any groove or bell (See Fig. 1).

Section 22(b).—Change "33 F" to read "40 F."

Figure 1.—Cut off the upper and lower bearing blocks at the inside edge of the groove, and delete the footnote. Label the lower bearing block "At least 6 in. wide."

Standard Specifications for Concrete Drain Tile (C 412 – 60):

Section 10(a).—Change to read as follows:

(*a*) For Standard-Quality concrete drain tile, the three-edge bearing crushing strength as shown in Table I, Column A, or the three-edge bearing crushing strength and the 5-hr boiling absorption as shown in Table I, Column B, shall meet the requirements given in Table I. No absorption tests are required if the strength requirements of Table I, Column A, are met.

Table I.—Change to read as shown in the accompanying Table III, retaining present footnotes.

Table III.—Change the heading which now reads: "Minimum Three-Edge Bearing Crushing Strength . . . " to read "Minimum Individual Three-Edge Bearing Crushing Strength[a], lb per lin ft."

Section 15(c).—Delete first sentence and add the following two sentences:

(*c*) No wall thickness is specified for Standard-Quality concrete drain tile where the crushing strength and the absorption tests are used to determine the tile quality. When only the crushing strength is used to determine Standard-Quality tile, then the wall thickness for Standard-Quality tile shall not exceed the nominal shell thickness given in Table II by more than 25 per cent.

In the present second sentence insert the words "at any point" between the words "tile" and "shall."

Section 19.—Delete Paragraph (*b*) and reletter Paragraph (*c*) as (*b*).

Section 23.—Add a new Paragraph (*d*) to read as follows:

(*d*) The average absorption for the Standard-Quality tile and for the Extra-Quality tile shall be the average of the absorption tests for the 5 tile constituting the standard sample as defined in Section 12, or the average of the absorption tests for the strongest and the weakest tile as measured by the crushing strength of the 5 tile of the standard sample. When drain tile fail to meet the absorption test requirement as computed by averaging the absorptions from the weakest and the strongest tile of a standard sample, then the average absorption test shall be computed by averaging the absorption tests from all the five tile from the standard sample. The average absorption for the Special-Quality tile shall be the average of the absorption tests for the 5 tile constituting the standard sample.

Tentative Revision of Standard

Standard Specification for Concrete Sewer Pipe (**C 14 – 59**):

Table II.—Change the wall thickness for 18-in. diameter pipe from "2 in." to "2¼ in." Also, change the wall thickness for 21-in. diameter pipe from "2½ in." to "2¾ in."

Editorial Change

Standard Specifications for Reinforced Concrete Low-Head Pressure Pipe (**C 361 – 61**):

Section 13(a).—Add under compression set in the tabulation "70 C (158 F) for 22 hr."

Section 13(b).—In Item (*4*) under compression set add: "Method B."

This report has been submitted to letter ballot of the committee, which consists of 50 voting members; 36 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

P. W. MANSON,
Chairman.

HOWARD F. PECKWORTH,
Secretary.

REPORT OF COMMITTEE C-15

ON

MANUFACTURED MASONRY UNITS*

Committee C-15 on Manufactured Masonry Units held two meetings during the year: on June 28, 1962, New York, N.Y., in conjunction with the Annual Meeting; and on Feb. 18, 1963, in Washington D. C. in conjunction with meetings of Committees C-7 on Lime and C-12 on Mortars for Unit Masonry.

The committee consists of 87 members, of whom 40 are classified as producers, 19 as consumers, and 28 as general interest members.

Committee C-15, in cooperation with Committees C-12 and E-6 on testing Building Constructions, sponsored a Symposium on Masonry Testing[1] which was held on June 28 and 29, 1962, in conjunction with the Annual Meeting in New York City.

The increasing production and sale of plastic-coated masonry units has brought about a need for specifications and methods of testing for such coatings. Committee C-15 has accordingly been asked to assume the responsibility for developing such standards. The Executive Subcommittee has accepted that responsibility on behalf of the committee and authorized the appointment of a new subcommittee whose objective will be the development of standards for plastic-coated masonry units. The new subcommittee has been designated as Subcommittee IV, and R. C. Valore, Jr., has accepted the chairmanship.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee C-15 requests approval of the following recommendations. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[2] The existing standard and tentatives referred to appear in their present form in the 1961 Book of ASTM Standards, Part 5.

New Tentative

Tentative Specifications for Chemical-Resistant Ceramic Tower Packings, as appended hereto.[3]

Revision of Standard Immediate Adoption

(requires nine-tenths affirmative vote)

Standard Specifications for Sewer Brick (Made from Clay or Shale) (**C 32 – 58**):

Section 2(b).—Change the second sentence to read: "Brick having absorptions not exceeding an individual maximum of 20 per cent shall be graded *MA* if the compressive strength conforms to the requirements for Grade *SA*."

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] Published as *ASTM STP 320.*

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[3] The new tentative appears in the 1963 Supplement to *Book of ASTM* Standards, Part 5.

Tentative Continued Vithout Revision

Tentative Method of Test for:

Drying Shrinkage of Concrete Block (**C 426 – 61 T**), and

Moisture Condition of Hardened Concrete by the Relative Humidity Method (**C 427 – 58 T**).

Tentative Specification for:

Vitrified Clay Filter Block for Trickling Filters (**C 159 – 61 T**).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Clay Brick and Structural Clay Tile (J. E. Neville, chairman) met twice during the year. Work was completed on a draft of a proposed Tentative Specification for Structural Clay Non-Load Bearing Screen Tile. It is currently undergoing a final review prior to submittal to the main committee for approval. Task groups working on such special studies as chippage and dimensional tolerance limitations for facing brick, durability of clay products, and clarification of brick specifications continued their work, but have not yet made their recommendations to the subcommittee. New task groups have been appointed to review the coring requirements for brick and the Standard Definitions of Terms Relating to Structural Clay Tile (C 43). The latter group has been instructed to consider the expansion of these definitions to include terms relating to all structural clay products.

Subcommittee III on Concrete and Sand Lime Units (H. T. Toennies, chairman) met twice during the year and is continuing to review proposed extensive revisions of the standards under its jurisdiction. The subcommittee hopes to submit some of these revisions to the main committee for action at the Annual Meeting in June, 1963.

Subcommittee IV on Plastic-Coated Masonry Units (R. C. Valore, Jr., chairman) held its first meeting in February. This meeting was devoted to a discussion of the scope of the subcommittee and the initial work it will undertake.

Subcommittee V on Water-Retardant Materials for Masonry Units (T. Ritchie, chairman).—After a year of relative inactivity due to a change in chairmen, this subcommittee met in February and reviewed its work to date in order to make plans for its resumption.

Subcommittee VI on Glazed Brick and Tile (E. G. Wanner, chairman) approved the adoption as standard of the Tentative Specifications for Ceramic Glazed Structural Clay Facing Tile, Facing Brick, and Solid Masonry Units (C 126).

Subcommittee IX on Chemical Resistant Units (W. W. Perkins, chairman) completed the proposed Tentative Specifications for Chemical-Resistant Ceramic Tower Packings appended to this report.[3]

This report has been submitted to letter ballot of the committee, which consists of 87 members; 64 members returned their ballots, of whom 61 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

F. A. BIBERSTEIN,
Chairman.

M. H. ALLEN,
Secretary.

REPORT OF COMMITTEE C-16

ON

THERMAL INSULATING MATERIALS*

Committee on C-16 on Thermal Insulating Materials and its subcommittees held two meetings during the year: in Ashville, N. C. on Sept. 24 to 27, 1962, and in Washington, D. C., on Feb. 24 to 27, 1963.

During the year, resignations were received from J. M. High as Secretary; from W. L. Scott as Member-at-Large to the Executive Subcommittee; and from John B. Mochel as Assistant Secretary. All of these resignations were accepted and the Executive Subcommittee elected as replacements: A. Grant Wilson, Division of Building Research, National Research Council, Ottawa, Canada, as Secretary; J. F. Corey, Pittsburgh Plate Glass, Pittsburgh, Pa., as Assistant Secretary; and W. C. Turner, Union Carbide Chemicals Co., South Charleston, W. Va. as Member-at-Large.

At the Annual Meeting in June, 1962, Charles B. Bradley, one of the charter members of this committee, was presented with the Award of Merit by the Board of Directors.

The committee recommended that all standards, except specifications, under their jurisdiction be submitted to the American Standards Assn. for approval as American Standards.

Due to the heavy work load of Subcommittee S-1 on Preformed Block and Pipe Insulation, it was decided to remove from this subcommittee all development of methods of tests and assign this work to Subcommittee T-VIII on General Standards. The by-laws were, therefore, changed to permit any Committee C-16 member to be a member of both S-1 and T-VIII plus one other subcommittee of their selection.

Subcommittee T-VII recommended to Committee C-16 that Method E 162 be recognized as suitable for measuring surface flammability of thermal insulating materials and finishes but not as a basis of ratings for building code purposes.

The committee consists of 99 voting members, of whom 48 are classified as producers, 25 as consumers, and 26 as general interest members. There are also 30 non-voting members, 1 associate member, and 6 honorary members.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1962 annual meeting, Committee C-16 presented to the Society through the Administrative Committee on Standards the following recommendations which were accepted on the dates indicated:

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

New Tentative Method of Test for:

Dry Adhesion of Thermal Insulating or Finishing Cement to Thermal Insulating Blocks (C 504 – 62 T) (Accepted December 17, 1962.)

Revision of Tentative Specification for:

Amosite Asbestos Thermal Insulation for Pipes (C 391 – 59 T) (Accepted September 14, 1962).

The new method and the revised specification appear in the 1962 Supplement to Book of ASTM Standards, Part 5.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee C-16 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] All of the existing tentatives and standards referred to appear in their present form in the 1961 Book of ASTM Standards, Part 5.

New Tentatives

as appended hereto[2]

Tentative Methods of Test for:

Thermal Conductivity of Materials by the Heat Flow Meter Method,
Density of Fibrous Loose Fill Building Insulations, and
Density of Granular Loose Fill Insulations

Tentative Specifications for:

Vermiculite Loose Fill Insulation, and
Diatomaceous Earth Block and Pipe Thermal Insulation.

Tentative Recommended Practice for:

Determining Nesting Pipe Sizes of Preformed Insulation for Pipes (IPS System).

Revision of Tentatives

as appended hereto[2]

Tentative Methods of Test for:

Water Vapor Transmission of Materials Used in Building Construction (**C 355 – 59 T**).

Tentative Specifications for:

Mineral Fiber Molded-Type Pipe Insulation for Elevated Temperatures (**C 281 – 58 T**),
Mineral Fiber Molded-Type Pipe Insulation for Low Temperatures (**C 300 – 52 T**),
Mineral Fiber Block or Board Thermal Insulation for Low Temperatures (**C 378 – 60 T**), and
Mineral Fiber Batt Insulation (Industrial Type) (**C 262 – 55 T**).

Revision of Standard, Immediate Adoption

(requires nine-tenths affirmative vote)

Standard Methods of Test for Thermal Conductivity of Materials by Means of the Guarded Hot Plate (**C 177 – 45**):

Revise as appended hereto.[2]

Mineral Fiber Block or Board Thermal Insulation for Elevated Temperatures (**C 392 – 60**):

Revise as appended hereto.[2]

Tentatives Continued Without Revision

Tentative Recommended Practice for:

Prefabrication and Field Fabrication of Thermal Insulation Fitting Covers (**C 450 – 60 T**).

Tentative Methods of Test for:

Mixing Thermal Insulating Cement Samples (**C 163 – 61 T**),
Breaking Strength and Calculated Modulus of Rupture of Preformed Insulation for Pipe (**C 446 – 59 T**), and
Corrosion Effect of Thermal Insulating Cements on Base Metals (**C 464 – 61 T**).

Tentative Specifications for:

Mineral Fiber Blanket and Felt Insulation (Industrial Type) for Low Temperatures (**C 264 – 60 T**),

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[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[2] The new and revised tentatives and the revised standards appear in the 1963 Supplement to *Book of ASTM Standards*, Part 5.

Mineral Fiber Blanket and Felt Insulation (Industrial Type) for Elevated Temperatures (**C 382 - 61 T**),
Mineral Fiber Thermal Insulating Cement (**C 195 - 60 T**), and
Mineral Fiber Hydraulic-Setting Thermal Insulating and Finishing Cement (**C 449 - 60 T**).

Withdrawal of Tentatives

Tentative Specification for:

Diatomaceous Earth Thermal Insulation for Pipes (**C 334 - 59 T**), and
Diatomaceous Earth Block-Type Thermal Insulation (**C 333 - 59 T**).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee S-1 on Preformed Block and Pipe Insulation (P. L. Losse, chairman).—Work is continuing on the development of material specification for polystyrene block and pipe covering in both the molded and extruded types. Also, specifications are in process for foamed polyurethane block and pipe covering. This work is recognizing the function of Committee D-20 on Plastics which committee is developing specifications for these products for all purposes other than thermal insulation.

This report has been submitted to letter ballot of the committee, which consists of 99 voting members; 74 members returned their ballots, of whom 72 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. C. LEWIS,
Chairman.

A. GRANT WILSON,
Secretary.

REPORT OF COMMITTEE C-17
ON
ASBESTOS-CEMENT PRODUCTS*

Committee C-17 on Asbestos-Cement Products and its subcommittees met twice during the year: on June 25 and 26, 1962, during the Annual Meeting of the Society in New York, N. Y., and on Jan. 17 and 18, 1963, in New York, N. Y.

The committee consists of 40 members, of whom 36 are voting members; 15 are classified as producers; 7 as consumers; 18 as general interest members; and 1 as consulting member.

During the year the committee lost 4 members through resignation or retirement and gained 4 members by letter ballot procedure.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1962 Annual Meeting, Committee C-17 presented to the Society, through the Administrative Committee on Standards, the following recommendations:

New Tentatives:

Specification for Asbestos-Cement Perforated Underdrain Pipe (C 508 – 63 T), and

Methods of Testing Asbestos-Cement Pipe (C 500 – 63 T).

Revision of Tentative Specifications for:

Asbestos-Cement Pressure Pipe (C 296 – 59 T), and

Asbestos-Cement Non-Pressure Sewer Pipe (C 428 – 59 T).

These recommendations were accepted by the Standards Committee on April 10, 1963 and will appear in the 1963 Supplement to the Book of ASTM Standards, Part 5.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee C-17 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee letter ballot on the recommendations will be reported at the Annual Meeting.[1] The existing tentatives referred to appear in their present form in the 1961 Book of ASTM Standards, Part 5.

Adoption of Tentative as Standard Without Revision

Tentative Methods of Sampling and Testing Asbestos-Cement Flat Sheets, Roofing and Siding Shingles, and Clapboards (**C 459 – 60 T**).

Tentative Definitions of Terms Relating to Asbestos-Cement and Related Products (**C 460 – 60 T**).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Research (G. J. Verbeck, chairman) has four projects under consideration and is developing detailed programs for (1) sulfate resistance of asbestos-cement products, (2) rapid method for degree of autoclave

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*** Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.**

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

cure, (3) nature of asbestos to cement bond, and (4) acid resistance of asbestos-cement products.

Subcommittee III, ISO/TC 77, on Asbestos-Cement Products (J. R. Cran, chairman) followed the activities of the ISO committee at its meetings in Milan, Italy, in October, 1962, and in Rome, Italy in May, 1963, with the subcommittee chairman as official representative.

Subcommittee IV on Roofing and Siding Shingles (F. A. Crippen, chairman) is considering further changes in specifications for asbestos-cement shingles and siding

Subcommittee V on Flat and Corrugated Sheets (W. A. Heausler, chairman) is preparing a draft specification for asbestos-cement insulating panels.

Subcommittee VI on Asbestos-Cement Pipe (C. R. Meek, chairman) has extracted the Methods of Testing Asbestos-Cement Pipe from the former Specifications and Methods C 296 and C 428, and has recommended their publication as a separate tentative. Included in the new Methods (C 500) is an uncombined calcium hydroxide test which replaces the present alkalinity test.

The subcommittee has revised the specifications for pressure pipe and non-pressure sewer pipe (C 296 and C 428) and has recommended a specification for asbestos-cement perforated underdrain pipe. These latest specifications classify the pipe into three types depending upon the requirements for uncombined calcium hydroxide. The amount of uncombined calcium hydroxide in type I pipe may not exceed 3 per cent, and in type II pipe may not exceed 1 per cent. There is no chemical requirement for type III pipe. The classification of types I and II corresponds to the requirements of Federal Specification SS-P-331b.

A task group has been appointed consisting of W. Price (chairman), E. Farrell, J. Mandry, and F. Mardulier, to study the question of broadening the description in the specifications of the uses of the three different types of pipe. Further, a request was made of Research Subcommittee II that it continue to study the problem of finding the most suitable test to determine the ability of asbestos-cement pipe to resist corrosive attack.

This report has been submitted to letter ballot of the committee, which consists of 33 voting members; 28 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. V. FRIEDLAENDER,
Chairman.

S. E. WILLIAMS,
Secretary.

REPORT OF COMMITTEE C-18

ON

NATURAL BUILDING STONES*

Committee C-18 on Natural Building Stones held one meeting during the year: on Feb. 5, 1963, in Montreal, Canada. Subcommittee II on Nomenclature and Definitions and Subcommittee IV on Specifications also met on Oct. 8 and 9, 1962, in Washington, D. C. The Advisory Subcommittee met on Feb. 22, 1963 in Washington, D. C.

The committee consists of 31 members, of whom 14 are classified as producers, 7 as consumers, 9 as general interest members, and 1 as honorary member. There are 4 consulting members.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1962 Annual Meeting, Committee C-18 presented to the Society through the Administrative Committee on Standards a new Tentative Specification for Exterior Marble. The specification was accepted by the Standards Committee on Jan. 4, 1963, and it appears in the 1962 Supplement to Book of ASTM Standards, Part 5, bearing the designation C 503 – 62 T.

RECOMMENDATIONS FOR SOCIETY ACTION

Committee C-18 requests approval by the Society at the Annual Meeting of the following recommendation:

Tentative Continued Without Revision

Tentative Method of Test for Combined Effect of Temperature Cycles and Weak Salt Solutions on Natural Building Stones (**C 218 – 48 T**).

SUBCOMMITTEE ACTIVITIES

Subcommittee IV on Specifications (F. S. Eaton, chairman).—Task groups for sandstone, limestone, and slate are preparing proposed specifications covering sandstone, dimension limestone, blackboard slate, and structural slate. A task group on granite is being reorganized for the purpose of continuing studies leading toward an improved specification for structural granite.

This report has been submitted to letter ballot of the committee, which consists of 31 members; 26 members returned their ballots, of whom 23 have voted affirmatively and 2 negatively.

Respectfully submitted on behalf of the committee,

F. S. EATON
Chairman.

W. F. MULLEN
Secretary.

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

REPORT OF COMMITTEE C-20
ON
ACOUSTICAL MATERIALS*

Committee C-20 on Acoustical Materials held two meetings during the year: in Philadelphia, Pa. on Oct. 24 to 26, 1962, and in North Aurora, Ill., on March 20 to 22, 1963.

The committee consists of 73 members, of whom 30 are classified as producers, 11 as consumers, and 32 as general interest members.

During the past year, the members of the committee were saddened to learn of the death of Howard Hardy.

The Board of Directors of the Society has approved a change in the scope of the committee, enlarging it to include sound transmission. A new Subcommittee VI on Sound Transmission has been formed with R. K. Cook as chairman. This subcommittee is identical in personnel with Subcommittee III of Committee E-6 on Methods of Testing Building Constructions. Standards prepared by this subcommittee will be promulgated by Committee E-6.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee C-20 requests approval by the Society at the Annual Meeting of the following recommendations. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] The existing tentative referred to appears in its present form in the 1961 Book of ASTM Standards, Part 5.

New Tentatives

as appended hereto.[2]

Tentative Methods of Test for:

Air Flow Resistance of Acoustical Materials, and

Light Reflectance of Acoustical Materials by the Integrating Sphere Reflectometer.

Tentative Continued Without Revision

Tentative Method of Test for Sound Absorption of Acoustical Materials in Reverberation Rooms (**C 423 – 60 T**).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Sound Absorption (H. J. Sabine, chairman) is studying the final draft of a new box method for measuring sound absorption. A study of the effect of temperature and humidity on sound absorption at high frequencies is also under way.

Subcommittee II on Flame Resistance (R. W. Boltz, chairman) continues the study of flame propagation on acoustical tile in the 8-ft and 25-ft tunnel tests, particularly with a view toward developing a rating for acoustical membrane materials. Active liaison with Committee E-5 on Fire Tests of Materials and Construction is being maintained.

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[2] The new tentatives appear in the 1963 Supplement to *Book of ASTM Standards*, Part 5.

Subcommittee III on Maintenance (W. M. Rees, chairman) is working on revision of a proposed standard on brush, spray, and roller painting of acoustical tile.

Subcommittee IV on Application (R. L. Lindahl, chairman) is working on a draft of a Recommended Practice for Acoustical Materials Suspension Systems. A draft has been circulated for comment to Committee C-20 members, acoustic tile manufacturers, suspension system manufacturers, members of National Acoustical Contractors Assn. and Acoustical Materials Assn., and non-member acoustical contractors.

Subcommittee V on Basic Physical Properties (W. A. Jack, chairman) is working on a new method for determining the air flow resistance of acoustical materials. This method is entirely different from the new tentative method appended to this report.

Subcommittee VI on Sound Transmission (R. K. Cook, chairman) is studying impact sound transmission in building structures and is also preparing a method of determining sound transmission in the field.

This report has been submitted to letter ballot of the committee, which consists of 73 members; 51 returned their ballots, of whom 50 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

R. N. HAMME,
Chairman.

RALPH HUNTLEY,
Secretary.

REPORT OF COMMITTEE C-21
ON
CERAMIC WHITEWARES AND RELATED PRODUCTS*

Committee C-21 on Ceramic Whitewares and Related Products held one meeting during the year: in New York, N. Y., on April 29, 1962.

The committee consists of 56 voting members, of whom 26 are classified as producers, 6 as consumers, and 24 as general interest members.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1962 Annual Meeting, Committee C-21 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Method of Test for:

Determining Facial Dimensions and Thickness of Flat, Rectangular Ceramic Wall and Floor Tile (C 499 – 62 T),

Relative Resistance to Wear of Unglazed Ceramic Tile by the Taber Abraser (C 501 - 62 T) and

Wedging of Flat, Rectangular Ceramic Wall and Floor Tile (C 502 - 62 T).

Tentative Methods C 499 and C 501 were accepted by the Standards Committee on July 27, 1962, and Tentative Method C 502 was accepted on Sept. 11, 1962. All appear in the 1962 Supplement to Book of ASTM Standards, Part 5.

RECOMMENDATIONS FOR SOCIETY ACTION

Committee C-21 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] All of the existing tentatives and standards referred to in this report appear in their present form in the 1961 Book of ASTM Standards, Part 5.

Tentative Revision of Standard

Standard Definitions of Terms Relating to Ceramic Whitewares and Related Products (**C 242 – 60**):

Add the following definitions:

Ceramics.—A general term applied to the art or technique of producing articles by a ceramic process, or to the articles so produced.

Ceramic Process.—The production of articles or coatings from essentially inorganic, nonmetallic materials, the article or coating being made permanent and suitable for utilitarian and decorative purposes by the action of heat at temperatures sufficient to cause sintering, solid-state reactions, bonding, or conversion partially or wholly to the glassy state.

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] No action was taken at the Annual Meeting on the Report of Committee C-21 in the absence from the session at which the report was scheduled for presentation of an individual authorized to present the report. Society action on the recommendation, set forth herein, was subsequently taken through the Administrative Committee on Standards. The tentative revision of Standard Definitions C 242 – 60 was accepted by the Standards Committee on Sept. 20, 1963.

Ceramic Article.—An article having a glazed or unglazed body of crystalline or partly crystalline structure, or of glass, which body is produced from essentially inorganic, nonmetallic substances and either is formed from a molten mass which solidifies on cooling, or is formed and simultaneously or subsequently matured by the action of the heat.

Moisture Expansion.—An increase in dimension or bulk volume of a ceramic article caused by reaction with water or water vapor.

NOTE.—This reaction may occur in time at atmospheric temperature and pressure, but is expedited by exposure of the article to water or water vapor at elevated temperatures and pressures.

Tentatives Continued Without Revision

Tentative Methods of Test for:

Bond Strength of Glazed Interior Ceramic Wall Tile in Portland Cement Installations (**C 482 - 61 T**),

Electrical Resistance of Conductive Ceramic Tile (**C 483 - 61 T**),

Thermal Shock Resistance of Glazed Ceramic Tile (**C 484 - 61 T**), and

Measuring Warpage of Ceramic Tile (**C 485 - 61 T**).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature (J. F. Day, chairman) has reviewed and recommends adoption of Tentative Definitions C 242 as standard. Four new definitions have been approved by the committee as recommended in this report.

Subcommittee II, Editorial (R. F. Geller, chairman) has thoroughly reviewed all of Committee C-21 standards and tentatives for editorial content. The subcommittee is awaiting final action on Methods C 329 and C 373 which require definitions or clarifications, by Subcommittee 12 of Committee E-1.

Subcommittee III on Fundamental Properties (J. T. Roberts, chairman) is continuing its work on developing test methods. Two test methods are being restudied in order to resolve negative ballots. These methods will be submitted as soon as possible.

Subcommittee V on Nuclear Applications (J. H. Handwerk, chairman) has continued its study of the utilization of ceramic materials in nuclear energy. These studies have indicated that the use of beryllia and urania in nuclear reactors has developed to a level where it is now possible to formulate standards, and a task group on beryllia has been established. The use of uranium oxide as fuel in thermal power reactors is well established; however, many parameters pertaining to fabrication methods, irradiation behavior, and maximum burnup of the fuel are still being investigated.

In addition to these two areas, interest in nonoxide ceramic materials such as carbide, nitrides, and sulfides for nuclear fuels is rapidly developing. These materials, however, have not been developed to the point where meaningful standards could be proposed. It is anticipated that standards may be applicable for one or more of these materials within the next two years, and, if warranted, task groups will be established to propose standards.

Ceramic materials, especially the fuel materials, have heretofore been studied for thermal reactors. In the expanding nuclear reactor technology, fast reactor concepts are receiving considerable interest. Thus, ceramic materials containing plutonium will be developed, and this will be another area which will require standards.

Subcommittee VI on Ceramic Tile (A. M. Illing, chairman) held a meeting on Dec. 6, 1962, at the Research Center of the Tile Council of America at Princeton, N. J., as well as during the regular meetings of Committee C-21. Four tentative test methods, mentioned earlier in the report, adopted in 1961 are recommended to continue as tentative for two more years. The three test methods accepted during the year by the Administrative Committee on Standards, referred to earlied in the report, were prepared by the subcommittee. A revised test method for bond strength of ceramic tile is being reviewed by the Editorial Subcommittee II. Other methods for ceramic tile are undergoing development by the subcommittee.

Task Group C on Non-Plastics (W. P. Shulhof, chairman) has completed its work on a suggested method for use of micromesh screens on Al_2O_3, SiO_2, and $CaCO_3$ powders and will shortly submit the method for committee examination. A method for the use of the MSA Particle Size Analyzer is now undergoing testing.

Respectfully submitted on behalf of the committee,

N. T. MORRISON,
Chairman.

R. C. WILSON,
Secretary.

REPORT OF COMMITTEE C-22
ON
PORCELAIN ENAMEL*

Committee C-22 on Porcelain Enamel held two meetings during the year: on Sept. 26, 1962, in Philadelphia, Pa., and on Feb. 7, 1963, in Montreal, Quebec, Canada, during ASTM Committee Week.

The committee consists of 46 members of whom 40 are voting members; 23 are classified as producers, 2 as consumers, and 15 as general interest members.

Because of the resignation of A. L. Friedberg as first vice-chairman, G. H. McIntyre was appointed to finish the unexpired term.

A more comprehensive name for the committee has been approved by committee letter ballot, namely, "Committee C-22 on Porcelain Enamel and Related Ceramic-Metal Systems." The new name is being submitted for approval of the Board of Directors. This expanded name will link the work of the committee more positively with related activities in other professional societies.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee C-22 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] The tentatives referred to appear in their present form in the 1961 Book of ASTM Standards, Part 5.

Adoption of Tentative as Standard With Revision

Tentative Definitions of Terms Relating to Porcelain Enamel (**C 286 - 61 T**):

Revised Definitions. — Change the definitions of Egg Shell, Firing, and Slip to read as follows:

Eggshell or Eggshelling.—The texture of a fired ceramic coating similar in appearance to the surface of an eggshell. In porcelain enamel, usually a defect.

Firing.—The controlled heat treatment of ceramic ware in a kiln or furnace to develop the desired final properties.

Slip (Slurry).—A suspension of finely divided ceramic material in liquid.

New Definitions.—Add new definitions to read as follows:

Set.—A flow property of porcelain enamel slip affecting the rate of draining, residual thickness, and uniformity of coating.

Adherence.—The degree of adhesion of a porcelain enamel or other ceramic coating to the metal substrate.

NOTE.—See the Method of Test for Adherence of Porcelain Enamel and Ceramic Coatings to Sheet Metal (ASTM Designation: C 313).

Bond.—See *Adherence.*

Cover Coat.—The layer of porcelain enamel normally applied over a ground coat.

Jumpers or Jumping.—See *Poppers.*

Tentative Revision of Standard

Add as a tentative revision to Definitions C 286, the revised definition of Pinhole and the new definitions of Glass-Coated Steel, and One-Coat Ware as follows:

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* Sixty-sixth Annual Meeting of the Society, June 23-28, 1963.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Pinhole, Pinholing.—Imperfections in the surface of a ceramic body or coating resembling pin pricks.

Glass-Coated Steel, Glass-Lined Steel, Glassed Steel.—A class of porcelain enameled steel which has high resistance to chemical attack at elevated temperatures.

One-Coat Ware, One-Coat Work.—(*1*) Articles finished in a single coat of porcelain enamel; (*2*) Sometimes a contraction of one-cover-coat ware, in which the finish consists of a single cover coat applied over ground coat.

Tentatives Continued Without Revision

Tentative Methods of Test for:

Acid Resistance of Porcelain Enamels (**C 282 – 61 T**), and

Spalling Resistance of Porcelain Enameled Aluminum (**C 486 – 61 T**).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Research (B. J. Sweo, chairman) is investigating the problems of protective coatings with regard to techniques of application, corrosion and erosion resistance, and effect of temperature.

Subcommittee II on Nomenclature (E. E. Howe, chairman) is continuing the study and preparation of definitions of terms relating to porcelain enamel and recommended the adoption with revision of Definitions C 286, as noted earlier in this report.

Subcommittee III on Education (M. J. Bozsin, chairman) has continued its contacts with the technical press and with the industry in general in view of increasing interest in Committee C-22.

Subcommittee IV on Materials (J. F. Matejczyk, chairman) has ten active projects under way. New projects include the composition and control of cast iron for porcelain enameling, metal pretreatment, and measurement of warp in porcelain enamel steel systems.

Subcommittee V on Finished Products (J. C. Richmond, chairman) has 13 active task groups and has prepared for committee approval two new methods for an image gloss test and a copper sulfate weathering test. The subcommittee recommended that Methods C 282 and C 486 be continued without revision.

This report has been submitted to letter ballot of the committee, which consists of 40 voting members; 33 members returned their ballots, of whom 26 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. N. HARRISON,
Chairman.

G. H. SPENCER-STRONG,
Secretary.

REPORT OF COMMITTEE C-24
ON
JOINT SEALANTS*

Committee C-24 on Joint Sealants held one meeting during the year, on June 28, 1962, in New York, N.Y., during the Annual Meeting of the Society.

The committee consists of 104 members, of whom 41 are classified as producers; 28 as consumers; 23 as general interest members; and 12 as consulting members.

During the year the committee approved editorial revisions in the American Standard Specification for Polysulfide-Base Sealing Compounds for the Building Trade (ASA No. A116.1–1960).

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee C-24 requests approval by the Society at the Annual Meeting of the following recommendations. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1]

New Tentatives

as appended hereto.[2]

Tentative Specification and Methods of Test for Expanded Elastomeric Preformed Gasket and Sealing Material, and

Tentative Method of Test for Staining and Color Change of One- or Two-Part Joint Sealants.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature (H. Lipkind, chairman) is continuing its work on establishing definitions of terms used in the specifications being prepared by the committee.

Subcommittee II on Bulk Compounds (F. Frybergh, chairman) held one meeting on June 28, 1962. The organization of the subcommittee is as follows:

Task Group I on Methods of Tests (B. Pozniak, chairman), with Subgroup A on Adhesion and Aging (B. Pozniak, chairman), Subgroup B on Application Life, and Subgroup C on Staining (A. Hockman, chairman). Task Group I has developed a staining test, and is developing a test for determining application life.

Task Group II on Specifications (F. Frybergh, chairman) has made editorial revisions of American Standard ASA No. 116.1.

Task Group III on One-Package Sealants (A. Tenzer, chairman) met

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[2] The new tentatives appear in the 1963 Supplement to *Book of ASTM Standards*, Part 5.

Sept. 18, 1962, to discuss development of test methods for one-package sealants.

Subcommittee III on Preformed Shapes (R. McKinley, chairman) held several meetings during the year, and is composed of Task Group A on Architectural Seals (D. E. Jackson, chairman), Task Group B on Underground Pipe Seals (F. Valenziano, chairman), and Special Task Force on Glazing Gasket Production (M. F. Torrence, chairman).

The subcommittee has under consideration proposed tentative specifications for neoprene structural glazing and panel gasket material, being developed by Task Group A.

This report has been submitted to letter ballot of the committee, which consists of 104 members; 77 members returned their ballots, of whom 63 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

W. A. OBERDICK,
Chairman.

J. R. PANEK,
Secretary.

REPORT OF COMMITTEE C-25

ON

CERAMICS FOR ELECTRONICS*

Committee C-25 has held two meetings during the past two years: in San Francisco, Calif., Oct. 26, 1961, and in Boston, Mass., on Oct. 10, 1962. Subcommittee VI held meetings at the Magnetics Conference in Phoenix, Ariz., on Nov. 15, 1961, and in Pittsburgh, Pa. on Nov. 12, 1962. The Executive Subcommittee held two meetings during the year: in New York, N.Y. on May 1, 1962, in conjunction with the annual meeting of the American Ceramic Society; and in Boston, Mass. on Oct. 10, 1962, with the meeting of the Electronics Division, American Ceramic Society.

The committee consists of 72 members, of whom 26 are classified as producers, 23 as consumers, and 23 as general interest members.

Due to the resignation of W. E. Hauth, Jr., Subcommittee VII is temporarily without a chairman.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

The committee requests approval by the Society at the Annual Meeting of the following recommendations. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1]

New Tentatives

as appended hereto.[2]

Tentative Methods of Test for:

Ferrimagnetic Resonance Linewidth and Gyromagnetic Ratio of Nonmetallic Magnetic Materials,

Complex Dielectric Constant of Nonmetallic Magnetic Materials at Microwave Frequencies,

Nonmetallic Magnetic Cores to be Used in a Coincident Current Memory with a Two-to-One Selection Ratio Operating Under Full Switching Conditions,

Saturation Magnetization of Saturation Induction of Nonmetallic Magnetic Materials, and

Compressive Strength of High-Strength Ceramic Materials.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I, Executive (J. L. Pentecost, chairman) held two meetings during the year. Reports had been received from each subcommittee chairman and plans were made for the Annual Meeting.

Subcommittee II on Classification and Nomenclature (R. M. Pinkham, chairman) reported that a classification chart and some definitions had been approved by the committee. Other definitions are being developed and will soon be ready for committee approval.

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[2] The new tentatives appear in the 1963 Supplement to *Book of ASTM Standards*, Part 5.

Subcommittee III on General Methods of Test (Robert S. DuFresne, chairman) is working on a density measurement method which will be submitted for committee approval. Electrical resistivity measurement techniques are under study and dielectric measurement techniques are being coordinated in conjunction with Committee D-9 on Electrical Insulating Materials.

Subcommittee IV on Electrical Insulating Materials (H. D. Taylor, chairman) has conducted an interlaboratory study of a compressive strength method, and the method was accepted by the subcommittee in coordination with Committee F-1 on Materials for Electron Tubes and Semiconductor Devices and Committee C-21 on Ceramic Whitewares and Related Products. A task group has been appointed to study thermal conductivity methods.

Subcommittee V on Ferroelectrics (W. W. Coffeen, chairman) is planning to develop a method for planer coupling coefficient of circular disks specimens. This is to be done by circulating a method for discussion, followed by an interlaboratory study for confirmation. Efforts have been made to stimulate studies on capacitor test methods since the task group on process controls was discontinued. There was also interest in developing a method for chemical analysis of barium titanate.

Subcommittee VI on Nonmetallic Magnetic Materials (D. L. Fresh, chairman) has been coordinating its activities with those of the Institute of Radio Engineers (IRE) and the American Institute of Electrical Engineers (AIEE) (now combined in Institute of Electrical and Electronics Engineers (IEEE)). Task group activities center around computer applications, microwave applications, and pulse transformer applications. Efforts are being made to establish suitable subjects for research projects for graduate research studies. To date, dielectric loss in garnets and square loop ferrites studies have been suggested.

Subcommittee VII on Semiconductor Materials, reported the need for study before significant work can be initiated by this group.

Subcommittee VIII on Composites (W. H. Liederbach, chairman) reviewed the proposed revised scope which brought to light three new task groups: on metallic coatings, on electrical contacts, and on hermetic seals. The current activities are concerned with test requirements on metal-ceramic packaging and solid state devices. The first task will deal with methods and specifications to establish metal-ceramic headers in conjunction with industry needs.

Subcommittee IX on Research (Gilbert Goodman, chairman) noted that 13 university ceramic departments had been polled on topics for graduate research in electronic ceramics. Committee C-25 membership has been solicited for suggested topics, and next year the Electronic Division of the American Ceramic Society will also be approached. Suggestions will be transmitted to the universities interested.

This report has been submitted to letter ballot of the committee, which consists of 69 voting members; 37 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. L. PENTECOST,
Chairman.

D. W. LUKS,
Secretary.

REPORT OF COMMITTEE D-1
ON
PAINT, VARNISH, LACQUER, AND RELATED PRODUCTS*

Committee D-1 on Paint, Varnish, Lacquer, and Related Products held two meetings during the year: on June 25 to 27, 1962, in New York, N. Y., in connection with the Annual Meeting of the Society, and in New Orleans, La., Jan. 7 to 9, 1963.

At the June, 1962, meeting, V. P. Miniutti, U. S. Department of Agriculture Forest Products Laboratory, Madison, Wis., presented a talk on "Wood Properties that Affect Coating Performance." Mr. Miniutti reviewed the earlier information obtained by the Forest Products Laboratory and others on the properties of wood that have a bearing on the subject, such as species, cell structure, density, grain, moisture content, knots, and extractives. He also discussed recent research conducted by him which has uncovered some new information on microstains that develop in wood profiles at the wood-paint interface.

An outstanding event of the January, 1963, meeting was a Symposium on Weathering, with G. L. Leithauser, General Motors Corp. as moderator. The following topics were presented and discussed:

"Exterior Weathering Tests—Their Purpose and Value," by Maurice Van Loo, Director of Paint Research, The Sherwin-Williams Co., Chicago, Ill.;

"Comparison of Replicate Florida and Delaware Exposure Results for High Gloss Enamels," by Mark P. Morse, Research Supervisor, Marshall Laboratories, E. I. duPont de Nemours and Co., Inc., Philadelphia, Pa.; and

"Effects of Different Environments on the Degradation of Paints," by W. E. Laesch, Supervisor of Florida Test Field, Polymer Department, General Motors Research Corp.

Committee D-1 has continued its close cooperation with the Federation for Paint Technology and as a result over 125 standards have been approved by both organizations. The Joint Federation-ASTM Committee on Paint, Varnish, and Lacquer (F. Scofield, chairman) serves as the permanent liaison between the two organizations.

Committee D-1 through its Committee on Intercommittee Relations (J. C. Weaver, chairman) has continued to maintain close contact with those technical committees whose activities are of interest to Committee D-1.

At the June, 1962, meeting, Honorary Memberships in Committee D-1 were granted to Louis A. Melsheimer and Armand E. Keeley. Also, at the June, 1962, meeting, W. T. Pearce, chairman of D-1, announced that on nomination from the Award of Merit Committee, the Board of Directors of the Society presented to Maurice Van Loo, one of Committee D-1's most faithful members, an ASTM Award of Merit.

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee D-1 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standards require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] All of the existing tentatives and standards referred to in this report appear in their present form in the 1961 Book of ASTM Standards, Part 8, or the 1962 Supplement.

New Tentatives

Tentative Methods of Test for:

Acetaldehyde Content of Vinyl Acetate,[2]
Purity of Acetaldehyde,[2]
Hydroquinone in Vinyl Acetate,[2]
Concentration of Formaldehyde Solutions,[2]
Pentaerythritol,[2]
Low Quantities of Mercury in Paint,[3]
Perspiration Resistance of Organic Coatings,[3, 4]
Adhesion of Coatings of Paint, Varnish, Lacquer, and Related Products With Scrape-Adhesion Apparatus,[3]
Rheological Properties of Non-Newtonian Materials,[3]
Slump of Oil and Resin-Base Caulking Compounds,[3]
Staining of Oil and Resin-Base Caulking Compounds,[3]
Stain Removal from Multicolor Lacquers,[3] and
Measurement of Plasticizer Migration from Vinyl Fabrics to Lacquers.[3]

Tentative Method for:

Preparation of Hot-Dipped Non-Passivated Galvanized Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products.[3]

Tentative Specifications for:

Vinyl Acetate,[2] and
Acetaldehyde.[2]

Tentative Recommended Practice for:

Testing Traffic Paints.[3]

Tentative Pictorial Surface Preparation Standards for:

Painting Steel Surfaces (these standards utilize the standards developed by the Swedish Standards Assn.)[3]

Revision of Tentative

Tentative Method of Test for Color of Transparent Liquids (Gardner Color Scale) (**D 1544 - 58 T**):
Revise as appended hereto.[5]

Revision of Standard and Reversion to Tentative

Standard Specifications for Raw Castor Oil (**D 960 - 52**):
Revise as appended hereto.[6]

Adoption of Tentatives as Standards Without Revision

Tentative Specifications for:

Distilled Coconut Fatty Acids (**D 1841 - 61 T**),
Distilled Corn Fatty Acids (**D 1842 - 61 T**),
Fractionated and Distilled Cottonseed Fatty Acids (**D 1843-61 T**), and
Asbestos-Cement Shingle Blanks to be Used as Panels in Weathering Tests of Latex and Emulsion Paints (**D 1911 - 61 T**).

Tentative Methods of Test for:

Hydroxyl Value of Fatty Oils and Acids (**D 1957 - 61 T**),
Color Change of White Architectural Enamels (**D 1543 - 60 T**),
Package Stability of Latex Paint (**D 1849 - 61 T**), and
Flash Point of Volatile Flammable Materials by Tag Open-Cup Apparatus (**D 1310 - 62 T**).

Tentative Methods for:

Qualitative Determination of Fish Oil in Drying Oils and Drying Oil Fatty Acids (**D 1724 - 60 T**),
Preparation of Aluminum Alloy Panels for Testing Paint, Varnish, Lacquer, and Related Products (**D 1733 - 60 T**),
Making and Preparing Concrete and Masonry

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[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[2] This new tentative appears in the *1964 Book of ASTM Standards*, Part 20.

[3] This new tentative appears in the *1964 Book of ASTM Standards*, Part 21.

[4] Discussion of this method is appended to the report; see pp. 325 and 328.

[5] The revised tentative appears in the *1964 Book of ASTM Standards*, Part 21.

[6] The revised tentative appears in the *1964 Book of ASTM Standards*, Part 20.

Panels for Testing Paint Finishes (**D 1734 – 60 T**),
Chemical Analysis of Dry Mercuric Oxide Pigment (**D 284 – 57 T**),
Chemical Analysis of Zinc Dust (Metallic Zinc Powder) (**D 521 – 50 T**),
Chemical Analysis of White Titanium Pigments (**D 1394 – 56 T**),
Chemical Analysis of Basic Lead Silico-Chromate (**D 1844 – 61 T**), and
Reporting Paint Film Failures Characteristic of Exterior Latex Paints (**D 1848 – 61 T**).

Revision of Standards
Immediate Adoption

(requires nine-tenths affirmative vote)

Standard Methods of Chemical Analysis of Dry Red Lead (D 49 – 44):

Section 10.—Change to read as follows:

10. Determine the amount of water soluble material present in accordance with the procedure in Section 3 of the Methods of Test for Common Properties of Certain Pigments (ASTM Designation: D 1208).

Standard Methods of Analysis of Barium Sulfate Pigments (D 715 – 45):

Sections 6 to 9.—Change to read as follows:

6. Determine the pH in accordance with the procedure in Section 4 of the Methods of Test for Common Properties of Certain Pigments (ASTM Designation: D 1208).

7. Determine the amount of water soluble material present in accordance with the procedure in Section 3 of the Methods of Test for Common Properties of Certain Pigments (ASTM Designation: D 1208).

8. Determine the moisture and other volatile matter in accordance with the Method of Test for Hydroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments (ASTM Designation: D 280).

9. Determine the coarse particles in accordance with the Methods of Test for Coarse Particles in Pigments, Pastes, and Paints (ASTM Designation: D 185).

Standard Methods of Analysis of Mica Pigments (D 716 – 45):

Sections 6 and 9.—Change to read as follows:

6. Determine the moisture and other volatile matter in accordance with the Method of Test for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments (ASTM Designation: D 280).

9. Determine the coarse particles in accordance with the Methods of Test for Coarse Particles in Pigments, Pastes, and Paints (ASTM Designation: D 185).

Standard Methods of Analysis of Magnesium Silicate Pigment (D 717 – 45):

Sections 4 to 6.—Change to read as follows:

4. Determine the loss on ignition in accordance with Section 2 of the Methods of Test for Common Properties of Certain Pigments (ASTM Designation: D 1208).

5. Determine the moisture and other volatile matter by the Method of Test for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments (ASTM Designation: D 280).

6. Determine the coarse particles in accordance with the Methods of Test for Coarse Particles in Pigments, Pastes, and Paints (ASTM Designation: D 185).

Section 7.—Delete this section, and renumber Sections 8 and 9 as Sections 7 and 8.

Standard Methods of Analysis of Aluminum Silicate Pigment (D 718 – 45):

Sections 2 and 5 to 7.—Change to read as follows:

2. Determine the silicone dioxide content in accordance with the procedure in Section 2 of the Methods of Analysis of Magnesium Silicate Pigment (ASTM Designation: D 717).

5. Determine the loss on ignition by the procedure in Section 2 of the Methods of Test for Common Properties of Certain Pigments (ASTM Designation: D 1208).

6. Determine the moisture and other volatile matter by the Method of Test for Hygroscopic Moisture (and Other Matter Volatile Under Test Conditions) in Pigments (ASTM Designation: D 280).

7. Determine the coarse particles in accordance with the Methods of Test for Coarse Particles in Pigments, Pastes, and Paints (ASTM Designation: D 185).

Section 8.—Delete this section, and renumber Sections 9 and 10 as Sections 8 and 9.

Standard Methods of Analysis of Diatomaceous Silica Pigment (D 719 - 51):

Sections 2 and 3.—Change to read as follows:

2. Determine the moisture and other volatile matter by the Method of Test for Hygroscopic Moisture (and Other Matter Volatile Under Test Conditions) in Pigments (ASTM Designation: D 280).

3. Determine the loss on ignition in accordance with Section 2 of the Methods of Test for Common Properties of Certain Pigments (ASTM Designation: D 1208).

Standard Method of Test for Viscosity of Transparent Liquids by Bubble Time Method (D 1545 - 60):

Revise as appended hereto.[7]

Standard Method of Test for Distillation Range of Lacquer Solvents and Diluents (D 1078 - 58):

Revise as appended hereto.[7]

Reapproval of Standards

Standard Methods of Test for:

Coarse Particles in Pigments, Pastes, and Paints (**D 185 - 45**),
Bleeding of Pigments (**D 279 - 31**),
Hygroscopic Moisture (And Other Matter Volatile Under the Test Conditions) in Pigments (**D 280 - 33**),
Chemical Analysis of Dry Cuprous Oxide and Copper Pigments (**D 283 - 52**),
Chemical Analysis of Zinc Yellow Pigment (Zinc Chromate Yellow) (**D 444 - 51**), and
Acetone Extract in Black Pigments (**D 305 - 51**).

Recommended Practice for:

Conducting Exterior Exposure Tests of Paints on Wood (**D 1006 - 56**).

Tentatives Continued Without Revision

Tentative Method for:

Chemical Analysis of Strontium Chromate Pigment (**D 1845 - 61 T**).

Tentative Methods of Test for:

Acidity in Lacquer Solvents and Diluents (**D 1613 - 61 T**),
Water-Soluble Matter in Iron Oxide Pigments by Specific Resistance Method (**D 1846-61 T**), and
Two Parameter, 60-Deg Specular Gloss (**D 1471 - 57 T**).

Editorial Changes

With the 1962 revision of the three specifications listed below, Committee D-1 had recommended that the words "for Use in Paint, Varnish, Lacquer, and Related Products" be deleted from the titles and scopes and that the specifications be put under joint jurisdiction of Committees D-1 and D-16 on Industrial Aromatic Hydrocarbons and Related Materials. Publication was withheld by Headquarters of the changes in title and scope since Committee D-16 had not concurred in the action. As indicated in the Annual Report of Committee D-16, these specifications have now been approved by Committee D-16 and it is recommending the withdrawal of its specifications for these products, namely, Specifications D 837, D 842, and D 844. Committee D-1 accordingly recommends that reference to paint, varnish, lacquer, and related products be deleted from the titles and scopes of the following three specifications and that they be published under joint jurisdiction of Committees D-1 and D-16:

Standard Specifications for:

Industrial 90 Benzene for Use in Paint, Varnish, Lacquer, and Related Products (**D 361-61**),
Industrial Grade Toluene for Use in Paint, Varnish, Lacquer, and Related Products (**D 362-61**), and
Industrial Grade Xylene for Use in Paint, Varnish, Lacquer, and Related Products (**D 364-61**).

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[7] This revised standard appears in the *1964 Book of ASTM Standards*, Part 20.

Standard Method of Sampling Liquid Oils and Fatty Acids Commonly Used in Paints, Varnishes, and Related Materials (D 1466 – 61):

Section 3(b).—Change to read as follows:

> 3 (*b*) *Zone Sampler*.—A 12-in. Curtin oil thief[3] shall consist of a graduated glass tube with either aluminum or cadmium-plated brass fittings.

Standard Method of Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments (D 1654 – 61):

Section 6(2).—In the tabular material under "Description of Failure," change "10" to "11" in line 7; "25" to "26" in line 8; "40" to "41" in line 9; and "60" to "61" in line 10.

Standard Method for Water Fog Testing of Organic Coatings (D 1735 – 62):

Note 7.—Change to read: "Recommended exposure periods are intervals of 24 hr to the total of 96, 192, 504, or 1008 hr."

Tentative Method of Test for Indentation Hardness of Organic Coatings (D 1474 – 62 T):

Section 2(d).—Delete the word "unrecovered" in the explanation of *l* and A_p.

Section 7(e).—Insert at the beginning of the first line the word "immediately." Change the second sentence to read: "Focus the microscope on the indentation so that both extremities of the long diagonal (that is, where the upper edges of the indentation just converge) are as sharp as possible."

Section 14(e).—In second line, change "Paragraph (*c*)" to read "Paragraph (*d*)", and in third line change "Section 12(*c*)" to read "Section 12(*a*)(*1*)."

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Drying Oils, (D. S. Bolley, chairman) completed its work on the new Tentative Specifications for Tall Oil Fatty Acids, a revision of the Specifications for Raw Castor Oil (D 960), and the new Tentative Methods of Test for Fatty Acid Composition by Gas Chromatography. A new group has been established to compare methods for determining color after heating of drying oils and fatty acids.

Subcommittee III on Bituminous Emulsions has been discharged by action of the Advisory Committee, and the activities delegated to Subcommittee XXIX on Painting of Metals.

Subcommittee IV on Traffic Paints (W. W. Burr, chairman) prepared the new Tentative Recommended Practice for Testing Traffic Paints. Work is under way on the development of a method for determining the night visibility of traffic paints.

Subcommittee V on Solvents (E. F. Rogers, chairman) prepared the revision of Method D 1078, and the new Methods of Test for: Acetaldehyde Content of Vinyl Acetate; Purity of Acetaldehyde; Hydroquinone Content of Vinyl Acetate; and Pentaerythritol. New tentative specifications have also been prepared for Vinyl Acetate and Acetaldehyde. Work is continuing on the evaluation of the government solvent specifications. A new task group has been established to develop a gas chromatographic method for determining methanol and formaldehyde in commercial formaldehyde solutions.

Subcommittee VI on Definitions (F. B.

[3] Manufactured by the W. H. Curtin Co., P. O. Box 118, Houston 1, Tex.

Stieg, chairman) has increased its membership through action of the Advisory Committee requesting each subcommittee to designate one person to be a member of Subcommittee VI and to represent his subcommittee in all matters relating to definitions. The committee has reviewed and commented upon the ASTM Word List for Glossary of ASTM Definitions.

Subcommittee VII on Accelerated Tests for Protective Coatings (N. B. Garlock, chairman) completed its work on the new Tentative Methods for Preparation of Galvanized Steel Panels for Testing Paint, Varnish, Lacquer and Related Materials. The new Tentative Method of Test for Perspiration Resistance of Organic Coatings was prepared in cooperation with Subcommittee XXV.[8] A recommended practice for conducting exterior exposure tests of paint on wood is being developed. Work is also in progress on procedures for testing industrial maintenance coatings for heat resistance and for testing appliance and automotive coatings for resistance to "overbake."

Subcommittee VIII on Methods of Chemical Analysis of Paint Materials (W. H. Madson, chairman) prepared the new Tentative Method of Test for Low Quantities of Mercury in Paint. Work is under way on the development of a test method for the determination of arsenic in the 0.1 per cent range. Progress has been made in the development of a test method for the chemical analysis of whole paint, the analysis of driers, and a revision of Method D 215.

Subcommittee IX on Varnishes (B. G. Brand, chairman) prepared the revisions of Methods D 1544 and D 1545. Cooperative work is under way on the development of test methods for determining in urethanes, free nitrogen, carbon, and oxygen, free diisocyanate content, and pot life. Interest for the development of a skinning test is being determined by a canvass of the membership of the committee.

Subcommittee X on Optical Properties (H. K. Hammond, chairman) has under consideration the revision of the definition of tinting strength and the possibility of using scattering coefficient as an index of tinting strength. The committee has formed a group to investigate methods for hiding power of colored paints and has invited interested persons who may not be members of Subcommittee X to join with them in this effort.

Subcommittee XII on Latex and Emulsion Paints (P. T. Howard, chairman) is considering the development of a method to determine resistance of latex paints to adverse effects of storage in contact with uncoated metal—for example, tin plate. A preliminary draft of recommended practices for the testing of latex and emulsion paints has been completed and circulated among members for comment.

Subcommittee XIII on Shellac has been discharged by action of the Advisory Committee, and the activities delegated to Subcommittee XI on Resins.

Subcommittee XIV on Statistical Application (M. P. Morse, chairman) has completed preliminary drafts for definitions, statements of precision, and presentation of test data for inclusion in the proposed recommended practice for evaluating test methods.

Subcommittee XV on Specifications for Pigments, Dry and in Oil (Peyton Wheeler, chairman) is preparing revisions of Specifications D 603, D 605, and D 607. The Subcommittee is considering a revision of the Specification for Titanium Dioxide Pigments (D 476) to bring it in line with the new Federal

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[8] For a statement on the development of this method, see p. 325.

Specification TT-P-42. Editorial changes in Method D 480 have been finalized, making it inclusive of both leafing and non-leafing types. Revision of Specifications for Aluminum Pigments (D 962) is also being considered.

Subcommittee XVII on Flash Point (H. E. Riley, chairman) is maintaining contact with the Paint Federation in its work on the flash point of paints. Method of Test for Flash Point by Tag Closed Tester (D 56) is being reviewed for possible revision.

Subcommittee XVIII on Physical Properties of Materials (J. P. McGuigan, chairman) prepared the new Tentative Methods of Test for: Adhesion of Coatings with Scrape-Adhesion Apparatus; and Rheological Properties of Non-Newtonian Materials. Work is under way on the development of suitable test methods for mar resistance and on the measurement of film thickness of organic coatings on nonmagnetic metal surfaces.

Subcommittee XIX on Putty, Glazing, and Caulking Compounds (H. L. Kelfer, chairman) prepared the new Tentative Methods of Test for: Slump of Oil and Resin Base Caulking Compounds; and Staining of Oil and Resin Base Caulking Compounds. The committee has some preliminary laboratory studies under way on a test method to determine tack-free time or "skin formation" of gun grade caulks. This test may also give some valuable information on paintability.

Subcommittee XX on Miscellaneous Raw Materials (K. E. Holt, chairman) has set up task groups to study methodology for different types of fatty nitrogen compounds.

Subcommittee XXV on Lacquers (R. F. Buller, chairman) prepared the new Tentative Methods of Test for: Measurement of Plasticizer Migration from Vinyl Fabrics to Lacquers; and Stain Removal from Multicolor Lacquers. A specification for wood lacquers is being developed by the Subcommittee and it is hoped that this specification will provide a guide for interpreting results of tests applicable to wood lacquers. Work is continuing on the suggested freezing-and-thawing stability tests for multicolor lacquers and a tentative method of test for particle size and distribution of multicolor lacquers by photographic standards.

Subcommittee XXIX on Painting of Metals (A. J. Eickhoff, chairman) has proposed that ASTM join with the Steel Structures Painting Council and the Swedish Corrosion Committee in sponsoring a joint set of pictorial standards for the surface preparation of steel prior to painting.

Provisional Subcommittee on Factory-Coated Wood Products (Francis Scofield, chairman) has completed its organization. Groups on Permeability of Coatings, Film Thickness, Moisture Blistering, and Durability have been formed and have work under way. In addition, three product-oriented working groups have been established. Their concern will be sampling procedures, applicability of developed test methods to specific industry products, and the stimulation of the development of test methods unique to these specific products.

This report has been submitted to letter ballot of the committee, which consists of 379 voting members; 217 members returned their ballots, of whom 186 have voted affirmatively, and 0 negatively.

Respectfully submitted on behalf of the committee,

W. T. PEARCE,
Chairman.

C. A. LOMINSKA,
Secretary.

APPENDIX

STATEMENT ON THE DEVELOPMENT OF METHOD OF TEST FOR PERSPIRATION RESISTANCE OF ORGANIC COATINGS[1]

PREPARED BY C. C. HIPKINS[2]

The following comments are offered in order to provide an understanding of the factors involved in evaluating the perspiration resistance of organic finishes.

It is axiomatic that perspiration does have a degrading effect on many types of organic finishes. Perspiration attack may be manifested in several different manners, such as dulling, discoloration, blistering, the softening of some coatings, the embrittlement of others and most often the reduction of adherence. Finishes on articles subjected to frequent handling may suffer such deteriorations unless their compositions have been selected to withstand perspiration attack. It follows that, to accomplish judicious selections, the evaluation of the durability of the formulated finishes for their resistance is a necessity. The problem posed is how can tests be devised to determine the effects of perspiration on organic coatings, preferably in an accelerated and controlled manner, so that the accuracy of the results will be, at least of an order, to make them meaningful and useful. This is not simply accomplished.

A consideration of the design of an appropriate test reveals a number of factors involved in such testing. First a decision has to be made as to whether natural or synthetic perspiration is to be used in the test method. This phase of the problem has led to a number of literature searches by a number of collaborators as to the composition of natural perspiration and also attempts to synthesize an artificial perspiration. Quite a volume might be written on this subject but an attempt will be made to briefly summarize the salient points, as discovered by investigators not only in this country but also by English and European scientists.

It is recognized that natural perspiration is not a definite compound. It is likely to contain a dozen or more constituents and varies in composition not alone from person to person but from time to time with the same individual. In fact, it is generally agreed that "freshly shed perspiration possesses an acid reaction and gradually becomes alkaline owing to bacterial action." In view of these accepted facts, questions arise as to what is to be established as a norm or a standard human perspiration and, lacking this, what of the composition of a synthetic perspirant for test purposes which would likely simulate the attack of the ever varying composition of the natural.

With this as a background, about 10 years ago, a task group of Subcommittee VII of Committee D-1 embarked on a round robin series of tests using synthetic perspirants for evaluating the perspiration resistance of four different finishes. The results were not fruitful.

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[1] See *1964 Book of ASTM Standards*, Part 21.

[2] Bell Telephone Laboratories, Inc., Murray Hill, N. J., chairman of Working Group on Chemical Resistance of Applied Enamels of Subcommittee VII on Accelerated Tests for Protective Coatings.

Subsequently, another group was formed from Subcommittee VII and XXV to investigate the possibility of employing a wearer type test. It was found that this test had been used by certain organizations in testing and selecting organic finishes for items frequently handled during their service life. Drawing upon their experience, a round robin testing program was devised. The collaborators included workers of both the Central and the Marshall Laboratories of DuPont, the National Paint, Varnish and Lacquer Association, Bell Telephone Laboratories, the Shell Chemical Corp., Sears Roebuck's Laboratory and the Rust-Oleum Corp. Four different types of finishes were selected for preparing the test specimens which 23 male individuals mounted on their garters and were in contact with the calf of the leg for 1 and 2 weeks. Physical tests such as hardness, abrasion resistance and scratch adhesion were conducted before and after exposure to natural perspiration to evaluate the changes in those properties resulting from the attack of perspiration. Changes in appearance and in resistance to certain chemicals were also observed. A report was prepared covering the latter chemical test and one consisting of 15 pages with attached 32 pages of tables and charts for the other phases. These reports were presented at a joint meeting attended by 27 members of Subcommittee VII and XXV, and based upon the results, it was decided to proceed with the preparation of a tentative method for testing the resistance of finishes to natural perspiration by the wearer method. The proposed specification was prepared, discussed, modified, where required, approved by the members of the two Subcommittees and finally by Committee D-1.

While the tentative method was in preparation, because of the ever present desire for a synthetic perspirant test, another series of tests were planned and conducted using the same finishes as above for preparing the test specimens and exposing them to two new and different perspirants. One of these was acidic and the other alkaline. Five laboratories cooperated in making the tests, which consisted of measuring the changes in appearance, corrosion protection and in hardness of the coatings upon exposure to the two perspirants for several periods of time. Others also were similarly tested after the wearer perspiration exposure.

Unfortunately, the results were so discouraging of the possibility of developing a synthetic perspirant test that the task force was discharged from further work. In view of the obvious desirability for a synthetic test in preference to the wearer test, it may be that the group will be reactivated at some future date. But this appears to be very doubtful unless and until some worker in the field develops a more promising synthetic perspirant that will more accurately produce the effects of natural perspiration on finishes.

With reference to the proposed tentative wearer method, several other points should be particularly emphasized. First, the method is not an absolute one but is comparative. This is necessary because of the recognized inconsistency of composition of natural perspiration. Consequently, the test is designed to always run a criterion finish to which the ones under test can be related in judging resistance to perspiration. This practice is similar to the desirable procedure of including a standard for comparison in outdoor weather testing at different times and in different locations, in view of the vagaries of the weather. (The latter has been adopted despite the inconsistencies of the degrading agent—

whimsical weather — and because a synthetic weather — accelerated weathering — has not been found sufficiently dependable to write into a method.) Secondly, the method is proposed as tentative and will not be adopted as standard until its worth is substantiated by the experiences gained through its use or it is suitably modified as dictated by the results of such experiences.

Those judgments support the acceptance of the wearer test method for evaluating the perspiration resistance of organic finishes as a tentative standard, at least until some better method is developed.

See additional comments on p. 328.

COMMENTS ON THE METHOD OF TEST FOR PERSPIRATION RESISTANCE OF ORGANIC COATINGS

By Sam Tour[1]

[The report of Committee D-1 as presented at the Annual Meeting contained a recommendation for the acceptance as tentative of a Method of Test for Perspiration Resistance of Organic Coatings. The following comments were made by Mr. Sam Tour in opposition to the acceptance of this recommendation.]

The Proposed Method of Test for Perspiration Resistance of Organic Coatings, being presented by Committee D-1 for publication by the Society as tentative, is essentially the same as the one presented in 1961 but referred back to the committee for further consideration.

Since no record was made of the objections I raised to the method at that time, it follows that the substance of my remarks went no further than the general open meeting at which they were made. To the best of my knowledge, the members of Committee D-1, the members of the Textiles Committee, the members of the Corrosion Committee and the members of the Society, as a whole, have not been advised of the objections raised.

Now Committee D-1 comes forward with the same method and offers no explanation or justification for what they now propose. A comparison of the present offering with the rejected offering of 1961 shows only one change and that is a minor one. In Section 3(*a*) they now want "five human wearers" to be used in each evaluation whereas two years ago they called for only three. In Section 6 they introduce two editorial changes. In Section 6(*b*) one day is defined as 24 hr. A marvelous addition that may be news to many members of Committee D-1 but I doubt that it is news to even a small percentage of ASTM members! In Section 6(*c*) they add that after the test specimens are evaluated after an exposure of at least 7 days, as per the 1961 version, thereafter they shall be evaluated "at least every 7 days." Is that a world shaking modification? With those modifications and with no further explanation or justification they have the effrontery to attempt to foist this rejected piece of nonsense on the Society.

Some of the faults, ambiguities, and absurdities of the method as proposed are given below in item sequence as written in the proposed method.[2]

2. (item). "Performance is graded by including a reference coating of known performance in the test." In ordinary performance testing of materials all tests should be run, at least, in duplicate. The inclusion of a single reference panel is inadequate.

3. (*a*) "A minimum of five human

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[1] Chemical and metallurgical engineer, New York, N. Y.

[2] See 1963 preprint of Report of Committee D-1 on Paint, Varnish, Lacquer, and Related Materials, p. 29.

wearers shall be used in the evaluation of each coating." In what age, weight, health, employment, sobriety, and activity brackets should they be? If women are included, is the inclusion independent of menstrual periods and of pregnancies? If unskilled laborers are to be included, is there no difference whether they are ditch diggers, farmers, janitors, or store clerks?

3. (*b*) "A belt or garter capable of handling at least four coated panels in contact with each wearer's skin is required." The test panels specified are $1\frac{1}{2}$ by 2 in. Four of these, with $\frac{1}{8}$-in. spacing between, cover a strip 2 in. wide by $6\frac{3}{8}$ in. long. Some difficulty will be encountered in having all four panels make equal contact around the calf of the leg of a small person. Of what material shall the belt or garter be made? May it be all rubber? Should it be textile covered? Should it be moisture proof? How wide shall it be? How tight must it be?

3. (*b*) Note 1. "When the coated panels are held against the body for long periods, the natural reaction is the opening of the skin pores and the exudation of perspiration which keeps the coating damp even in cool weather." Does the medical profession agree with this? Since when does pressure against an area of the skin cause "the opening of skin pores" and not the decreasing of the blood supply and the lowering of activity in that area? Just what "skin pores" does the committee wish to have open? Are they after the oil gland pores or the sweat gland pores or both? May it be that the committee has not learned that there are more things in the skin than sweat glands?

3. (*b*) Note 1. "The panels may be supported against the calves of the legs or the waist." What is wrong with the thighs? Much more space is available on the thighs than on the calves of the legs. What portions of the body in the vicinity of the waist shall be used? The belly or the small of the back? What is wrong with using the hips?

3. (*b*) Note 1. "As many as four different coatings may be evaluated at one time when they are supported in this manner." With four coated panels in the belt or garter and one of these a reference panel only three others remain, or can't the committee count that far? Using only one panel as a test of a coating only three coatings may be evaluated, not four. The idea that evaluations are to be based on single panels should be abhorrent to many people skilled in the art of testing.

5. Conditioning. "Test specimens shall be conditioned for 24 hr minimum at standard conditions etc." That is just dandy, but how is the prospective wearer or his or her calves or waist to be conditioned? Shall they be washed with Ivory soap or with Dial or pumice soap? Shall they be dusted with emery, sand or borated talcum powder?

6. (*b*) "The wearer shall not remove panels in any one day of 24 hr for more than $\frac{1}{2}$ hr." Must the wearer remove them when he takes a bath? May he take a bath during the $\frac{1}{2}$-hr period? Again, what restrictions are there to the kind of soap, the kind of powder or the kind of skin lotion the wearer might use? Should the panels be wiped, washed, or left untouched during this half hour rest period? Should they be returned to the exact same positions in the same sequence after each rest period or should they be randomized?

8. Report (*4*). "Methods of exposure of panels to human perspiration including date panels were attached to the wearer." If it makes a difference whether the method used is the "leg calf-garter" or the "waist-belt" method, why permit both? If it makes no difference why record and report? If the date on the

calendar makes a difference, surely the weather makes a difference. Why not require a daily record of the temperature and humidity?

8. Report (*5*). "Length of time and number and sex of individuals participating." Here, it seems, the committee admits that sex makes a difference but in item 3 (*a*) they did not. How inconsistent can they be?

This discussion would not be complete without some information on the nature of perspiration.

The following quotations are from volume 6 of *Medical and Health Encyclopedia* edited by Morris Fishbein, M.D., and published by H. S. Stuttman Co. 1959:

> Perspiration is about 98 to 99 per cent water.
>
> People vary as to the speed with which they being to perspire and as to the amounts of salts and water that they lose from the body during perspiration.
>
> People vary also as to the amount of perspiration from various portions of the body.
>
> The odor of the sweat varies according to the part of the body from which it is secreted. Ordinarily it is acid, but after profuse perspiration it tends to be neutral or alkaline.
>
> Perspiration is increased by heat, by drinking of warm drinks, by psychic causes such as fright and anxiety and by a number of drugs which are known to increase the flow of water from the skin.
>
> Perspiration is decreased by external cold and by various drugs which have been proved capable of decreasing perspiration.
>
> A number of special diseases affect the sweat glands.
>
> Often perspiration is affected by the emotional state.
>
> There is another condition in which sweat fails to be secreted by the body. This is exceedingly rare.
>
> In a third type of case there is an offensive odor to the sweat.
>
> In another condition the sweat is colored.

The above quotations represent information that is modern. Nagel's *Handbuch der Physiologie des Menchen*, published in 1907 in German contains considerable information on perspiration. The following is taken from that publication:

> The composition of sweat which is given on page 406, of Volume two, of this Work, varies somewhat depending upon the method by which it is obtained. The specific gravity varies from 1.001 to 1.010. The reaction is generally acid. With the increase in sweating, caused by heat, etc., the acid may be diminished. This acid is probably produced by the fatty acids present in the skin to keep it soft, but secreted by the sebaceous glands and not the sweat glands. The amount of solid matter varies from 4.4 g. to 22.6 g. per liter: the most important constitutent is obviously sodium chloride. There are also traces of calcium chloride, phosphates and sulphates of the alkalies, and a minute trace of iron oxide. A trace of magnesium is also present. The organic substances, fat cholesterin and fatty acids are present. Also traces of proteins, traces of creatinine, aromatic oxy acids, skatoxyl sulphuric acid and indoxyl sulphuric acid. Traces of uric acid may be present. About half of the organic material is made up of urea.

In commenting in 1931 on the above an eminent doctor of the Institute of Cancer Research stated as follows:

> Of course sweat varies a great deal in different persons, and in conditions of disease may be much more acid than in normal persons. I imagine that if you make up a solution containing the following parts, per thousand, you would have a pretty close approximation to sweat:

sodium chloride	5.2
calcium phosphate	0.2
magnesium phosphate	0.1
sulphuric acid	0.6
potassium hydrate	0.5

> To this should be added minute quantities of oleic and butyric acids. I do not know whether any of the aromatic oxy acids are purchaseable in the market, but I think that the butyric and oleic would cover the acid side of the sweat.
>
> As I said above, the reaction varies greatly

in different people and I do not think you need be too fussy about putting in quite a little of these acids. I have seen a man whose sweat would take the gun-metal finish off a watch in about five minutes: he must have been nearly as acid as battery fluid.

The September 22, 1934, issue of *Chemical Age*, p. 255, refers to investigations of sweat as carried out by Dr. C. C. N. Vass and Professor McSwiney at Leeds (England). It states that their report contains much information concerning the various substances such as urea, glucose, and sodium chloride found in sweat and that the average pH of fresh perspiration is 5.6 whereas after incubation at 37 C (body temperature) for several hours the perspiration (owing to bacterial changes causing an increase in ammoniacal nitrogen) attains a pH of about 7.8. They go on to suggest two synthetic perspiration test liquors as follows:

A. Acid Test Liquor:

2.95 g common salt
0.75 g urea
1000 ml water

Brought to pH 5.6 by the addition of 0.1 *N* acetic acid.

B. Alkaline Test Liquor:

0.839 g ammonium chloride
1.732 g common salt
0.424 ml 0.880 ammonia solution
1000 ml water

Brought to pH 7.8 by addition of 0.1 *N* acetic acid.

Just in case Committee D-1 wishes to use some other formulations for perspiration, they might turn to those that were in ASTM Method D 627– 41 T as formerly sponsored by Committee D-13 on Textile Materials.[3] These requirements in Methods of Test for Evaluating Compounds Designed to Increase Resistance of Fabrics and Yarns to Insect Pests" give two other formulations for synthetic perspiration as follows:

Acid Solution:

Dissolve 10 g of NaCl, 1 g of lactic acid and 1 g of disodium orthophosphate in distilled water and dilute to one liter.

Alkaline Solution:

Dissolve 10 g of NaCl, 4 g of U.S.P. $(NH_4)_2CO_2$ and 1 g of disodium orthophosphate in distilled water and dilute to one liter.

In later years it appears that ASTM Committee D-13 decided to leave these matters of the use of perspiration in testing textiles to the American Association of Textile Chemists and Colorists. ASTM Methods D 627 has been discontinued and reference is made to the Technical Manual of the AATCC—Method 28-1952.

What more information does Committee D-1 on Paint, Varnish, Lacquer, and Related Products need to be satisfied that its "Belly Band Test" is ridiculous?

In closing may I say that, in my opinion, this proposed standard is without merit and should not be accepted by the Society. If it is accepted I fear that it will hold the Society up for ridicule. To avoid this I move it be not accepted.

[Mr. W. T. Pearce, chairman of Committee D-1. in support of the approval of the method, called attention to the work that had gone into its development and to the fact that the method had been used in a number of recognized laboratories. The Method of Test for Perspiration Resistance of Organic Coatings was accepted for publication as tentative and will appear in the *1964 Book of ASTM Standards*, Part 21, bearing the designation D 2204 – 63 T.]

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[3] 1955 Book of ASTM Standards, Part 7, p. 121.

REPORT OF COMMITTEE D-2
ON
PETROLEUM PRODUCTS AND LUBRICANTS*

This report contains the recommendations of Committee D-2 on Petroleum Products and Lubricants based upon the recommendations of its subordinate groups.

Proposed Methods to be Published as Information

The following proposed methods have been recommended by subordinate groups of Committee D-2 for publication as information.[1] They have not been approved by Committee D-2 or by the Society.

Appendix I. Simulative Recirculating System Testing of Aerospace Hydraulic Fluids.
Appendix II. Viscosity of a Functional Fluid for Aerospace Application.
Appendix III. Field Method for Sampling Aerospace Fluids in Containers.
Appendix IV. Sampling Liquids from Aerospace Components.
Appendix V. Particulate Contamination of Hydraulic Fluids by Gravimetric Analysis.
Appendix VI. Identification of Metallic and Fibrous Contaminants in Aerospace Fluids.
Appendix VII. Vapor-Liquid Ratio of Gasoline.
Appendix VIII. Interconversion of the Analysis of C_5 and Lighter Hydrocarbons to Gas-Volume, Liquid-Volume, or Weight Basis.
Appendix IX. Moisture in Liquefied Petroleum (LP) Gases (Valve Freeze Method).
Appendix X. Preliminary Examination of Hydraulic Fluids (Wear Test).
Appendix XI. Aromatics on Light Naphthas, Reformates, and Gasolines by Gas Chromatography.
Appendix XII. Evaluation of White Mineral Oils by Ultraviolet Absorption.
Appendix XIII. Weight of Surface Wax on Waxed Paper.
Appendix XIV. Salt in Crude Oil (Electrometric Method).
Appendix XV. Calculating Viscosity Index. (proposed revision of Method D 567.)
Appendix XVI. Analysis of Oil-Soluble Petroleum Sulfonates by Liquid Chromatography.
Appendix XVII. Particulate Contaminant in Aviation Fuels.
Appendix XVIII. Determining Knock Characteristics of Liquefied Petroleum (LP) Gases by the Motor Method.
Appendix XIX. Detection of Hydrogen Sulfide in Liquefied Petroleum (LP) Gases (Lead Acetate Test).
Appendix XX. Vapor Forming Index (VFI) of Motor Gasolines.
Appendix XXI. Kinetic Coefficient of Friction for Wax Coatings.
Appendix XXII. Rapid Combustion Methods for the Determination of Sulfur in Liquid and Gaseous Hydrocarbon Materials (Wickbold and Beckman Burner Techniques).
Appendix XXIII. Proposed Viscosity System for Industrial Fluid Lubricants.

The following recommendations have been approved by Committee D-2 in accordance with the regulations of the Society. All of the existing tentatives and standards referred to appear in their present form in the 1961 Book of ASTM Standards, Part 7, or the 1962 Supplement thereto, unless otherwise noted.

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* **Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.**

[1] Appendixes I to VII, X, XI, XV, XVII, XX, and XXIII appear in the *1964 Book of ASTM Standards*, Part 17.

Appendixes VIII, IX, XII, XIII, XIV, XVI, XVIII, XIX, XXI, and XXII appear in the *1964 Book of ASTM Standards*, Part 18.

New Tentatives

Tentative Methods of Test for:

Autoignition Temperature of Liquid Petroleum Products,[2] as published in Appendix IX, 1960 Compilation of "ASTM Standards on Petroleum Products and Lubricants," Vol. I, p. 1073, with a revised Fig. 1 as shown in the accompanying Fig. 1.[3]

Smoke Density in the Flue Gases From Distillate Fuels,[4] as published in Appendix I, 1959 Compilation of "ASTM Standards on Petroleum Products and Lubricants," p. 1127, with Section 1 changed to read as follows:

1. *Scope.*—This method is designed to evaluate the smoke density in the flue gases from burning distillate fuels. It is intended primarily for use with home heating equipment burning kerosine or heating oils. It may be used in the laboratory or in the field to compare fuels for clean burning or to compare heating equipment.

Effect of Air Supply on Smoke Density in Burning Distillate Fuel,[4] as published in Appendix I, 1960 Compilation of "ASTM Standards on Petroleum Products and Lubricants," Vol. I, p. 1044, with the title changed as listed above, and with Section 1 changed to read as follows:

1. *Scope.*—This method is designed to evaluate the performance of distillate fuels from the standpoint of clean, efficient burning. It is intended primarily for use with home heating equipment burning kerosine or heating oils. It may be used either in the laboratory or in the field to compare fuels using a given heating unit or to compare the performance of heating units using a given fuel.

NOTE 1.—This method applies only to pressure atomizing and rotary type burners.

Residue in Liquefied Petroleum (LP) Gases (End Point Index Method)[2] as published in Appendix XIII, 1962 Compilation of "ASTM Standards on Petroleum Products and Lubricants," Vol. I, p. 1260, with Section 8 changed to read as follows:

8. *Precision.*—The following criteria should be used for judging acceptability of results for oil numbers 0 to 100 and residue numbers in the range of 0 to 60:

(*a*) *Repeatability.*—Duplicate results by the same operator should not be considered suspect unless they differ by more than the following amounts:

Oil Number Range	Repeatability
0 to 20........	4
20 to 40........	6
40 to 100........	8

Residue Number Range	Repeatability
0 to 20........	5
20 to 40........	10
40 to 60........	20

(*b*) *Reproducibility.*—The results submitted by each of two laboratories should not be considered suspect unless the two results differ by more than the following amounts:

Oil Number Range	Reproducibility
0 to 20........	6
20 to 40........	8
40 to 100........	12

Residue Number Range	Reproducibility
0 to 20........	10
20 to 40........	10
40 to 60........	30

Naphthenes in Saturates Fractions by Refractivity Intercept, as appended hereto.[4]

Analysis of Liquefied Petroleum (LP) Gases by Gas Chromatography, as appended hereto.[2]

Water and Sediment in Crude Oils (ASTM-API Method), as appended hereto.[4,5]

Specifications for Special Duty Propane, as appended hereto.[4]

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[2] This new tentative appears in the *1964 Book of ASTM Standards,* Part 18.

[3] Figure 1 is not included in this report but appears in the new tentative; see *1964 Book of ASTM Standards,* Part 18.

[4] This new tentative appears in the *1964 Book of ASTM Standards,* Part 17.

[5] Action was taken subsequent to the Annual Meeting to adopt this tentative as standard through the Interim Procedure.—ED.

Conversion of Kinematic Viscosity to Saybolt Universal Viscosity or to Saybolt Furol Viscosity, as appended hereto.[2]

Basic Calibration of Master Viscometers and Viscosity Oil Standards, as appended hereto.[2]

Thermal Stability of Hydraulic Fluids, as appended hereto.[2]

Revisions of Tentatives

D 396 - 62 T, Specifications for Fuel Oils:

Section 1(a).—Reference a new note to the word "specifications," and add a note to read: "For information on the significance of the terminology and test methods used in these specifications, see the Appendix."

Appendix.—Add an Appendix on the significance of the specifications, as appended hereto.[6]

D 611 - 55 T, Aniline Point and Mixed Aniline Point of Petroleum Products and Hydrocarbon Solvents:

Note 1.—Change the second sentence to read as follows: "... to ASTM Method D 611 - 51 T, the apparatus described in the Institute of Petroleum Method IP 2/52, and commercially available automatic aniline point apparatus are adequate... "

Section 5(a).—Insert immediately preceding Table I, the following new Note 3, renumbering subsequent notes:

NOTE 3.—The aniline point of aniline and *n*-heptane determined with automatic apparatus shall be 156.7 ± 0.4 F (69.3 ± 0.2 C) when corrected in accordance with the equation in Section A6 (*a*).

Section 7.—Change the first word "Two" to read "Three." Add a new paragraph to read: "*Method 3*, applicable when using automatic apparatus in accordance with instructions in the Appendix."

Appendix.—Add a description of Method 3 to read as follows:

METHOD 3

Apparatus

A5. *Automatic Aniline Point Apparatus,* commercially available, using a modified thin-film technique, and direct heating of the sample with electrical immersion heater. Detection of change of sample turbidity at the aniline point is by response of a photoelectric cell to collimated light directed through the thin film of sample.

Procedure

A6. (*a*) Determine the automatic aniline point in accordance with instructions provided with the apparatus. Correct the aniline point using the following formula:

$$\text{Corrected aniline point} = \frac{X_a - A}{B}$$

where:

X_a = automatic aniline point, and

A and B = constants determined for each apparatus as described in Paragraph (*b*).

NOTE 8.—It has been established by cooperative tests that observed aniline points determined by some automatic apparatus are lower than the determinations by Methods 1 and 2. The difference is greater for automatic apparatus when relatively high sample-cooling rates are used, and increases as the aniline point increases.

(*b*) Determine the aniline point by either Method 1 or Method 2 using the automatic apparatus for three or more samples with aniline points in each of the ranges 110 to 120 F, 140 to 150 F, and 170 to 180 F. Calculate A and B by the least squares method by simultaneous solution of the following equations:

$$\Sigma(X_a) = NA + B\Sigma(X_c)$$

$$\Sigma(X_aX_c) = A\Sigma(X_c) + B\Sigma(X_c^2)$$

where:

$\Sigma(X_a)$ = sum of all aniline point data by automatic apparatus,

$\Sigma(X_c)$ = sum of all aniline point data by either Method 1 or 2,

$\Sigma(X_c^2)$ = sum of the squares of all aniline point data by either Method 1 or 2,

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[6] The appendix is not included in this report but appears in the revised tentative; see *1964 Book of ASTM Standards,* Part 17.

$\Sigma(X_aX_c)$ = sum of the products of aniline point by either Method 1 or 2 using the automatic apparatus for each sample, and

N = number of samples.

NOTE 9.—Cooperative data were obtained from five laboratories for five samples with aniline points in the range 93 to 188 F. Constants A and B were calculated for the composite data as 0.79 and 0.991, respectively. Although a minimum number of nine samples is specified in this method, constants A and B in the preceding equation may be obtained with slightly greater precision if data for a larger number of samples are used.

D 614 – 60 T, Knock Characteristics of Aviation Fuels by the Aviation Method:[7]

The following applicable sections appear in ASTM Manual for Rating Aviation Fuels by Supercharge and Aviation Methods:

Section 112(a).—Change the fifth sentence and add a new sentence to read: "The carburetor intake is fitted with a curved pipe which is connected to the induction surge pipe assembly. The induction surge pipe assembly is preferred equipment; however, a surge tank may be used if desired."

TABLE I.—DETAILED REQUIREMENTS OF AVIATION TURBINE FUELS.
(Changes in Table I of Specifications D 1655)

Property	Jet A	Jet A-1	Jet B
Gravity, max, deg API (min, sp gr)	51 (0.7753)	51 (0.7753)	57 (0.7507)
Gravity, min, deg API (max, sp gr)	39 (0.8299)	39 (0.8299)	45 (0.8017)
Distillation temperature, deg F (deg C):			
10 per cent evaporated, max	400 (204.4)	400 (204.4)	...
20 per cent evaporated, max	...	...	290 (143.3)
50 per cent evaporated, max	450 (232.2)	450 (232.2)	370 (187.8)
90 per cent evaporated, max	...	...	470 (243.3)
Final boiling point, max, deg F (deg C)	550 (287.8)	550 (287.8)	...
Flash point, min, deg F (deg C)	110 (43.3)	110 (43.3)	...
Flash point, max, deg F (deg C)	150 (65.6)	150 (65.6)	...
Pour point, max, deg F (deg C)	−40 (−40)	...	...
Freezing point, max, deg F (deg C)	−40 (−40)	−58 (−50)	−60 (−51.1)
Viscosity at −30 F (−34.4 C) max, cs	15	15	...
Net heat of combustion, min, BTU per lb	18 400[c]	18 400[c]	18 400[c]
Copper Strip Corrosion:			
3 hr at 122 F (50 C), max	No. 1	No. 1	...
2 hr at 212 F (100 C), max	...	...	No. 1
Thermal stability at 300 to 400 F (148.9 to 204.4 C)[f]			

[c] Use for Jets A and A-1 the value calculated from Table IV or Eqs 4 and 5 in Method D 1405, Estimation of Net Heat of Combustion of Liquid Petroleum Products. Use for Jet B the value calculated from Table II or Eqs 2 and 5 in Method D 1405. Method D 240, Test for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter may be used as an alternative. In case of dispute, ASTM Method D 240 must be used.

[f] Thermal stability test shall be conducted for 5 hr at 300 F (148.9 C) preheater temperature, 400 F (204.4 C) filter temperature, and at a flow rate of 6 lb per hr.

Section 113.—Change the first sentence to read: "To maintain the inlet air temperature within specified limits, a heater is required either with the induction surge pipe assembly or with the surge tank when the humidity control apparatus is used."

Section 114.—Change the third sentence of the third paragraph to read: "The outlet pipe connects through a bend to the pipe leading to the induction surge pipe assembly which contains a heater for controlling the intake air temperature."

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[7] See *ASTM Manual for Rating Aviation Fuels by Supercharge and Aviation Methods,* 1958, and the 1961 Supplement thereto.

Section 505.—Change the last sentence to read: "For convenience, the induction surge pipe assembly and ice tower should be left until later."

Section 509.—Change the words "surge tank" to read "induction surge pipe assembly," in each of the four places this occurs in the section.

D 910 – 62 T, Specifications for Aviation Gasolines:

Add Centigrade equivalent temperatures to the specifications, as follows, other provisions remaining unchanged:

Table I.—Under "Requirements for All Grades":

Distillation temperature, deg F (deg C):	
10 per cent evaporated, max. . .	158 (70)
50 per cent evaporated, max. . .	221 (105)
90 per cent evaporated, min. . . .	212 (100)
90 per cent evaporated, max. . .	257 (125)
Final boiling point, max, deg F (deg C).	338 (170)
Sum of 10 and 50 per cent evaporated temperatures, min, deg F (deg C).	307 (152.8)
Freezing point, max, deg F (deg C).	−76 (−60)

Section 9(i).—Add "(100 C)" after "212 F."

D 1655 – 62 T, Specifications for Aviation Turbine Fuels:

Section 2.—Change "*Type A*," "*Type B*," and "*Type A-1*" to read "*Jet A*," "*Jet B*," and "*Jet A-1*."

Table I.—Add Centigrade equivalent temperatures and include new nomenclature "Jet" instead of "Type," as shown in the accompanying Table I. Those items not listed remain unchanged.

D 1656 – 62 T, Knock Characteristics of Motor Fuel Above 100 Octane Number by the Research Method:[8]

Section 6(m).—Change to read as shown below for Section 6(*m*) of Method D 908.

Section 12(a).—Change the third sentence to read: "The difference in antiknock level between the reference fuels selected for the final bracket shall not exceed 1.3 octane numbers in the 100 to 105 octane range or two octane numbers above 105 octane number."

Section 12(d).—Change the first and sixth sentences to read as follows:

> Make another reference fuel blend which it is expected will bracket the knockmeter reading of the sample, and which differs from the first reference fuel by no more than 1.3 octane numbers in the 100 to 105 octane range or two octane numbers above 105 octane number.
>
> If the readings on the reference fuel blends do not meet the foregoing requirement, make a third reference fuel blend differing by no more than the previously specified octane number difference from the reference fuel blend with the reading closest to the sample.

The following applicable sections appear in ASTM Manual for Rating Motor Fuels by Motor and Research Methods:

Section 314(d).—Change to read: "(*d*) Prepare two reference fuel blends, differing by no more than the specified octane number bracket for the octane number level involved, which will bracket the sample, that is, one reference fuel of higher and the other of lower octane number than the sample."

Sections 112(a), 113, 114, 301, 302, 303, 408(a), 505(a), 509, and new Figs. A and B.—Change to read as shown below for the same sections of Method D 357.

D 1659 – 59 T, Maximum Fluidity Temperature of Residual Fuel Oil:

Appendix.—Add a new Appendix entitled "Test for Fluidity of a Residual Fuel Oil at a Specified Temperature" to read as follows:

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[8] See *ASTM Manual for Rating Motor Fuels by Motor and Research Methods*, 1960, and the 1961 Supplement thereto.

Scope

A1. This method covers the determination of the fluidity of a residual fuel oil at a specified temperature in an as-received condition.

Significance

A2. This method may be used as a "go-no-go" procedure for operational situations where it is necessary to ascertain the fluidity of a residual oil under prescribed conditions in an as-received condition. The conditions of this method simulate those of a pumping situation where the oil is expected to flow through a ½-in. pipe under slight pressure at a specified temperature.

Outline of Method

A3. A sample of fuel in its as-received condition is cooled at the specified temperature for 30 min in the standard U-tube, and is tested for movement under prescribed pressure conditions.

Definition

A4. A sample is considered "fluid at the temperature of the test (degrees Fahrenheit)," if it will flow 2 mm in 1 min in a 12.5 mm U-tube under a maximum pressure of 15.2 cm of mercury.

Apparatus

A5. Identical to that described in Section 4 of Method D 1659, with the exception that ASTM Thermometer 1F, referred to in Section 4(*b*); Cold Bath, Section 4(*c*); and Cooling Baths, Section 4(*d*) are unnecessary.

Preparation of Apparatus

A6. Identical to that procedure described in Section 5 of Method D 1659.

Procedure

A7. (*a*) Pour the sample as received into a thoroughly cleaned and dry standard fluidity U-tube, without contacting the upper walls of the tube, until the vertical height of the sample in the U-tube is 1.5 in. Insert in one leg of each U-tube an ASTM Thermometer 5F, in a cork that has been grooved to permit the passage of air. The thermometer must be placed in the center of the tube and its bulb immersed so that the beginning of the capillary is 0.1 in. below the surface of the oil.

(*b*) Fix the tube in the bath set at the specified temperature, immersed to a depth of approximately 3 in. Control the bath and sample temperatures within ±2 F and ±1 F respectively, of the specified temperature of the test.

(*c*) After the sample has been at the specified temperature for 30 min, attach the U-tube to the automatic vacuum controller with the stopcock or pinchclamp, whichever is used, open. Wind the thread on the pulley attached to the synchronous motor. Turn the power switch to the ON position. Apply suction automatically to the U-tube at the prescribed rate. Observe any movement of the oil during a 1-min interval, the time required to obtain 15.2 cm of mercury vacuum over the oil in the U-tube. Immediately disconnect the U-tube from the automatic vacuum controller, turn off the power switch, and rewind the thread. If the oil moves at least 2 mm during the minute the suction is applied, the oil is considered fluid at the temperature of the test.

Report

A8. Report the fluidity of the sample at a specified temperature as follows:

(*1*) If the sample fulfills the conditions of flow, as defined in Section A4, report fluidity: "Fluid at (temperature of test in deg Fahr)"; or fluidity at (temperature of test in deg Fahr): "Pass."

(*2*) If the sample does not fulfill the conditions of flow, as defined in Section A4, report fluidity: "Not Fluid at (temperature of test in deg Fahr)"; or fluidity at (temperature of test in deg Fahr): "Fail."

Precision

A9. In the event that results on two samples of the same fuel oil do not agree, that is, one sample "passes" and one sample "fails" at the temperature specified for the test, make two additional determinations using ASTM Method D 1659, Test for Maximum Fluidity Temperature of Residual Fuel Oil, and apply precision data in accordance with Section 10(*b*) of Method D 1659.

D 1660 – 61 T, Thermal Stability of Aviation Turbine Fuels:

Footnote 4.—Change to read: "A single sheet of No. 12 Whatman paper is specified for this purpose."

Section 8.—Change to read as follows:

8. *Rating Preheater Deposits.*—Rate the preheater deposits using the tube deposit rating code on the ASTM Color Standard according to the following procedure:

(*a*) Dry the tube with a stream of pentane or hexane or by gently dipping in a non-agitated bath for a few seconds and hanging vertically in still air. Perform this operation immediately after shutdown and removal of the preheater and filter assemblies and carry out the tube rating within 30 min of tube removal.

(*b*) Visually examine the dry preheater tube under a diffused light source in a Tuberator. Each inch of tube length should be separately rated in the sequence noted in Fig. 1. When a section of the tube corresponds visually to a Color Standard, that number should be recorded. If the section being rated is in the obvious transition state between any two adjacent Tuberator Color Standards, it should be rated as less than the darker (that is, higher number) standard.

(*c*) In rating the preheater tube, the darkest deposits govern and the code number reported should be representative of the darkest section rather than average deposits. Small individual spots, streaks, or bands of dark deposits should be ignored provided the area of each such spot or streak is less than 0.05 sq in. (which corresponds to a circle about 0.25 in. in diameter). If there are more than three such areas on the entire tube, a fresh fuel sample shall be retested if it passes in all other respects. If a streak covering two or more adjacent sections of the tube causes a fail result, the test should be repeated on a fresh fuel sample since such a streak suggests misalignment of the heater element within the preheater tube. If a continuous spot, streak, or band falls on the boundary of sections being rated but covers a total area of 0.05 sq in. or more, it should be rated.

Note 8.—Delete present Note 8 and add a new Note 8 to read "It is important to ensure that all light bulbs in the Tuberator are functioning, because a change in light intensity can shift the rating standard significantly."

New Section 10.—Add a new Section 10 on Precision to read as follows:

The method is used generally in assessing fuel stability at a given temperature. Marginal stability is recognized most commonly by wide differences in the measurements of filter pressure differential.

All revisions of the method were tested by ten laboratories during the final phase of cooperative evaluations. Two determinations at 350 F preheater temperature on the same sample of a refined kerosine known as Bayol D differed from each other by the following amounts (95 per cent probability):

	Filter Differential Pressure, inches mercury	Tube Deposit Rating
Repeatability	0.2	1
Reproducibility	0.5	2

New Section 2.—Add a new Section 2 on "Significance" to read as follows, renumbering present Section 2 and subsequent sections accordingly:

2. *Significance.*—The test is indicative of fuel performance during jet aircraft operation and can be used to assess fuel stability at a given temperature by means of a pass-fail criterion.

Figure 1.—Under "Preheater Tube Deposit" add "(Section 9(*b*) and Tuberator, Appendix II, Section B25)".

D 1948 – 62 T, Knock Characteristics of Motor Fuels Above 100 Octane Number by the Motor Method:

Sections 6(n), 112(a), 113, 114, 301, 302, 303, 408(a), 505(a), 509, and New Figs. A and B.—Change to read as shown below for the same sections of Method D 357.

Sections 12(a), 12(d), and 314(d).—Change to read as shown above for the same sections of Method D 1656.

Adoption of Tentatives as Standard With Revision

D 447 – 59 T, Distillation of Plant Spray Oils:

Section 5(c).—Change to read as follows:

(*c*) When the percentage distilled at a certain temperature is required and the barometric pressure varies from 760 mm by more than 10 mm, use the temperature corrected to 760 mm prior to the distillation (Note 4).

NOTE 4.—Instead of correcting the temperature prior to the distillation, it is permissible to plot the final corrected temperatures against the corresponding percentages and report the indicated percentage at the required temperature from the curve.

Section 6.—Change to read as follows:

6. *Precision.*—The following criteria should be used for judging the acceptability of results (95 per cent probability):

(*a*) *Repeatability.*—Duplicate results by the same operator should not be considered suspect unless they differ by more than the following amounts:

	Repeatability
Thermometer reading at specified per cent recovered:	
10 per cent	4 F
50 per cent	3 F
90 per cent	4 F
Per cent recovered at 636 F	2 per cent

(*b*) *Reproducibility.*—The results submitted by each of two laboratories should not be considered suspect unless the two results differ by more than the following amounts:

	Reproducibility
Thermometer reading at specified per cent recovered:	
10 per cent	9 F
50 per cent	7 F
90 per cent	9 F
Per cent recovered at 636 F	4 per cent

D 483 – 61 T, Unsulfonated Residue of Petroleum Plant Spray Oils:

Section 10(a).—Under "Range of Unsulfonated Residue, per cent," change "85 to 96" to read "79 to 97."

Adoption of Tentatives as Standard Without Revision

D 322 – 58 T, Dilution of Gasoline-Engine Crankcase Oils,
D 482 – 59 T, Ash from Petroleum Oils,
D 808 – 57 T, Chlorine in New and Used Petroleum Products (Bomb Method),
D 874 – 59 T, Sulfated Ash from Lubricating Oils and Additives,
D 892 – 58 T, Foaming Characteristics of Lubricating Oils,
D 909 – 60 T, Knock Characteristics of Aviation Fuels by the Supercharge Method,
D 1264 – 53 T, Water Washout Characteristics of Lubricating Greases,
D 1478 – 57 T, Low-Temperature Torque of Ball Bearing Greases,
D 1548 – 59 T, Vanadium in Navy Special Fuel Oil,
D 1658 – 59 T, Carbon Number Distribution of Aromatic Compounds in Naphthas by Mass Spectrometry, and
D 1745 – 60 T, Calculation of Absolute Viscosity.

Withdrawal of Standards

D 446 – 53, Conversion of Kinematic Viscosity to Saybolt Universal Viscosity,
D 666 – 57, Conversion of Kinematic Viscosity to Saybolt Furol Viscosity,
D 810 – 59, Sulfated Residue, Lead, Iron, and Copper in Lubricating Oils, and
D 939 – 54, Saponification Number by Potentiometric Titration.

Methods D 446 and D 666 are being replaced by the new Tentative Method for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity or to Saybolt Furol Viscosity, as appended to this report. Methods D 810 and D 939 are being withdrawn due to lack of use.

Revision of Standards, Immediate Adoption

(requires nine-tenths affirmative vote)

D 127 – 60, Standard Method of Test for Melting Point of Petrolatum and Microcrystalline Wax, revised as appended hereto.[9]

D 357 – 62, Knock Characteristics of Motor Fuels of 100 Octane Number and Below by the Motor Method:[8]

Section 6(n).—Change the heading to read "Basic Cylinder Height Setting." Delete the last three sentences and add the following two sentences:

> The basic cylinder height setting is made by setting the micrometer at 0.352 or the digital counter at 930 when a compression pressure of 176 lb gage is obtained at a barometric pressure of 29.92 in. The compression pressures for other barometric pressures and other details of the procedure for obtaining and checking this setting are described in Sections 302 and 303.

The following applicable sections appear in ASTM Manual for Rating Motor Fuels by Motor and Research Methods:

Section 112(a).—In the 1961 Supplement to the 1960 Manual, the fifth sentence in this section was replaced by two sentences. Change the new fifth and sixth sentences to read as follows:

> The carburetor is fitted with a 90-deg elbow and vertical pipe which is connected to the induction surge pipe assembly, and they are the preferred equipment. The curved intake

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[9] This revised standard appears in the *1964 Book of ASTM Standards*, Part 18.

pipe and a surge tank are also approved equipment that may be used together or in various combinations with the preferred equipment.

Section 113—Change the first sentence to read:

> To maintain the inlet air temperature within specified limits, a heater is required either with the induction surge pipe assembly or the surge tank when the humidity control apparatus is used, or with the short-tube silencer when the humidity control apparatus is not used.

Delete the fifth and sixth sentences of this section and replace them with the following sentence: "The induction surge pipe assembly was specifically designed to make it easier to apply this insulation and to increase its effectiveness."

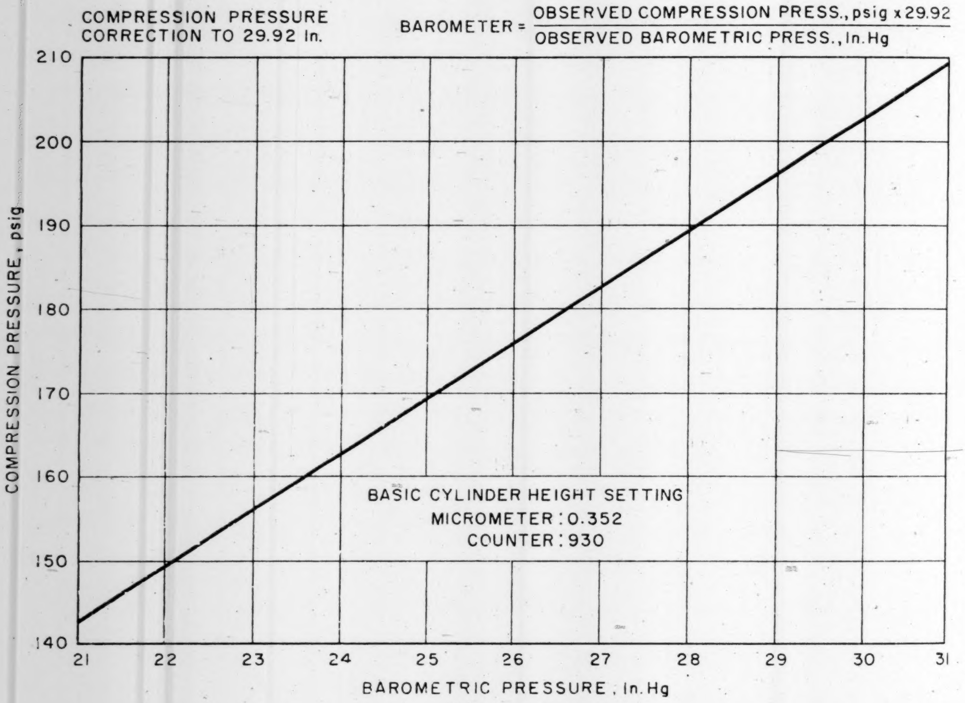

FIG. 2.—Compression Pressure Correction for Barometric Pressure (Based on Air Density) for Research Methods D 908 and D 1656.
(New Fig. A applicable to Methods D 908 and D 1656.)

Section 114.—Change the third sentence of the third paragraph to read: "The outlet pipe connects through a bend to the pipe leading to the induction surge pipe assembly which contains a heater for controlling the intake air temperature."

Section 301.—Change the title to "Basic Cylinder Clearance Volume." Delete the last three sentences of this section as given in the 1961 Supplement to the 1960 Manual and add the following sentences:

> Compression pressure in conjunction with a $\frac{5}{8}$-in. plug gage is used to obtain and check the basic cylinder clearance volume. When the basic cylinder clearance volume is established, the micrometer or digital counter is then set to indicate the compression ratio.

Section 302.—Change the title to Setting the Basic Cylinder Height.” Delete all the present paragraphs of this section and add the following two new paragraphs and new Figs. A and B as shown in the accompanying Figs. 2 and 3:

(*a*) *Research Methods:*

(*1*) With the engine at normal operating temperature, install a compression pressure gage equipped with check valve assembly 0105033 (as described in Waukesha Motor Co. Service Bulletin No. 77, Aug. 1, 1956) This check valve cartridge is fitted with Teflon wafer seals.

(*2*) If unit is equipped with a barometric compensator, it shall be set at 29.92.

(*3*) Adjust cylinder height to obtain the compression pressure indicated in Fig. A for the barometric pressure prevailing at the time of the test.

(*4*) Without changing the cylinder height setting obtained in Paragraph (*3*), adjust the indicator to the following reading:

Micrometer	0.352
Digital Counter	930

(*b*) *Motor Methods:* (Paragraphs (*1*), (*2*), and (*4*) same as Research Methods above):

(*3*) Adjust cylinder height to obtain the compression pressure indicated in Fig. B for the venturi size being used and for the barometric pressure prevailing at the time of the test.

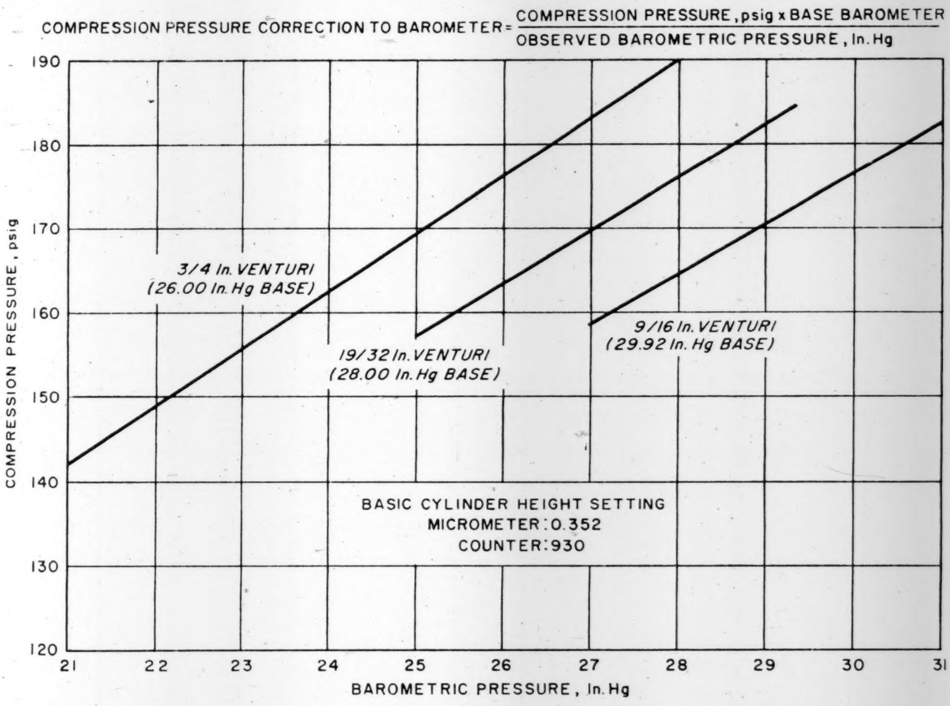

FIG. 3.—Compression Pressure Correction for Barometric Pressure (Based on Air Density) for Motor Methods D 357 and D 1948.

(New Fig. B applicable to Methods D 357 and D 1948.)

Section 303.—Change the title to “Checking Basic Cylinder Height.” Delete all the present paragraphs in this section and add the following new paragraphs:

303. The micrometer setting obtained in Section 302 is checked in the following manner:

(*1*) With the piston approximately 90 deg btdc on the firing stroke, raise the cylinder head to a micrometer reading of 0.400 (863 on digital counter) and insert a $\frac{5}{8}$-in. plug gage (Waukesha

Motor Co. Part No. A110546-A) through the pickup hole. Position the plug gage in the space between the valve heads and raise the piston to tdc. Lower the cylinder as required until the plug gage fits snugly between the piston head and the cylinder head. Record the cylinder height indication obtained.

Caution.—If the unit is equipped with a motorized compression ratio changer, do not move cylinder downward with plug gage in combustion chamber without first rotating flywheel to move piston at least 30 deg off dead center.

(*2*) Place the plug gage under the intake valve head and with the piston at tdc on the firing stroke adjust cylinder height until a snug fit is obtained between valve head and piston head. Record cylinder height reading obtained.

(*3*) Repeat Step (*2*) for the exhaust valve.

(*4*) Subtract the micrometer reading recorded in Step 1, from the micrometer reading obtained for each valve, Steps (*2*) and (*3*), to determine the amount each valve protrudes into the combustion chamber. The digital counter reading obtained for each valve shall be subtracted from the counter reading obtained in Step (*1*).

(*5*) Add together the differences obtained in Step (*4*) and multiply the sum (either micrometer or counter) by 0.156. This gives the cylinder height correction required to compensate for valve head volume in the combustion chamber.

(*6*) When the cylinder is equipped with a Champion No. 813 spark plug, *subtract* the Step (*5*) correction from 0.366 (the sum of the 0.352-in. basic setting and the 0.014-in. average valve head height correction).

When the cylinder under test is fitted with a Champion UD-16 spark plug, *subtract* the Step (*5*) correction from 0.362 (the sum of the 0.352-in. basic setting, the 0.014-in. average valve head height correction and a -0.004-in. correction for the smaller volume of the UD-16 spark plug).

When the cylinder is fitted with a Champion No. 813 spark plug, *add* the Step (*5*) correction to 910 (the basic counter setting of 930 minus the 20 unit average valve head correction).

When the cylinder is equipped with a Champion UD-16 spark plug, *add* the Step (*5*) correction to 916 (the basic counter setting of 930 minus the 20 unit average valve head correction plus a 6 unit correction for the UD-16 volume difference).

(*7*) The engine is unsatisfactory for testing fuels if the corrected micrometer value obtained in Step (*6*) is not within $+0.015$ or -0.010 in. of the cylinder height obtained with the $\frac{5}{8}$-in. plug gage in Step (*1*).

The corrected digital counter value obtained in Step (*6*) shall be within $+14$ or -21 units of the cylinder height indication obtained in Step (*1*).

NOTE.—The cylinder height indicator will require resetting if any component exposed to the combustion chamber is replaced by a different type component resulting in a volume change. The same setting values are used for both standard and oversized cylinders.

Section 408(a).—Change the first two sentences to read as follows:

> Compression pressure determined using the equipment described in Section 302 is an excellent check of general engine conditions. Deviations of 5 lb or greater from the pressures, shown in Table XXI for the Motor Method and Table XXII for the Research Method, indicate that maintenance is necessary.

Section 505(a).—Change the last sentence to read: "For convenience, the induction surge pipe assembly and ice tower should be left until later."

Section 509.—Change "surge tank" to read "induction surge pipe assembly" in each of the four places this occurs in this section.

D 908 – 62, Knock Characteristics of Motor Fuels of 100 Octane Number and Below by the Research Method:[8]

Section 6(m).—Change the title to "Basic Cylinder Height Setting." Delete the last three sentences and add the following two sentences:

> The basic cylinder height setting is made by setting the micrometer at 0.352 or the digital counter at 930 when a compression pressure of 202-lb gage is obtained at a barometric pressure of 29.92 in. The compression pressures for other barometric pressures and other details of the procedure for obtaining and checking this setting are described in Sections 302 and 303.

The following applicable sections appear in ASTM Manual for Rating Motor Fuels by Motor and Research Methods:

Sections 112(a), 113, 114, 301, 302, 303, 408(a), 505(a), 509, and New Figs. A and B:

Change to read as shown above for the same sections of Method D 357.

Tentatives Continued Without Revision

The committee has reviewed the following tentatives which have stood two or more years without revisions, and, for good and sufficient reasons discussed within the committee, recommends their continuation as tentatives without revision:

D 156 – 53 T, Saybolt Color of Petroleum Products (Saybolt Chromometer Method),
D 240 – 57 T, Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter,
D 381 – 61 T, Existent Gum in Fuels by Jet Evaporation,
D 721 – 56 T, Oil Content of Petroleum Waxes,
D 893 – 60 T, Insolubles in Used Lubricating Oils,
D 1018 – 55 T, Hydrogen in Petroleum Fractions,
D 1021 – 61 T, Oxygen in Light Hydrocarbon Vapors,
D 1022 – 59 T, Peroxide Content of Light Hydrocarbons,
D 1085 – 57 T, Gaging Petroleum and Petroleum Products,
D 1158 – 59 T, Bromine Number of Petroleum Distillates by Color-Indicator Titration,
D 1220 – 59 T, Calibrating Upright Tanks,
D 1317 – 57 T, Chlorine in New and Used Lubricants (Sodium Alcoholate Method),
D 1318 – 59 T, Sodium in Residual Fuel Oil (Flame Photometric Method),
D 1319 – 61 T, Hydrocarbon Types in Liquid Petroleum Products by Fluorescent Indicator Adsorption,
D 1320 – 60 T, Tensile Strength of Paraffin Wax,
D 1321 – 61 T, Needle Penetration of Petroleum Waxes,
D 1322 – 59 T, Smoke Point of Aviation Turbine Fuels,
D 1367 – 55 T, Lubricating Qualities of Graphites,
D 1405 – 61 T, Estimation of Net Heat of Combustion of Liquid Petroleum Products,
D 1465 – 57 T, Blocking Point of Paraffin Wax,
D 1477 – 57 T, Freezing Point of Aviation Fuels,
D 1479 – 57 T, Emulsion Stability of Soluble Cutting Oils,
D 1500 – 58 T, ASTM Color of Petroleum Products (ASTM Color Scale),
D 1550 – 58 T, ASTM Butadiene Measurement Tables,
D 1555 – 58 T, Calculation of Volume and Weight of Benzene, Toluene, and Paraxylene,
D 1661 – 59 T, Thermal Stability of U.S. Navy Special Fuel Oil,
D 1717 – 61 T, Analysis of Certain Light Hydrocarbons by Gas Chromatography,
D 1740 – 60 T, Luminometer Numbers of Aviation Turbine Fuels,
D 1831 – 61 T, Roll Stability of Lubricating Grease,
D 1832 – 61 T, Peroxide Number of Petroleum Wax,
D 1833 – 61 T, Odor of Petroleum Wax,
D 1834 – 61 T, 20-deg Specular Gloss of Waxed Paper,
D 1835 – 61 T, Specifications for Liquefied Petroleum (LP) Gases,
D 1836 – 61 T, Specifications for Commercial Hexanes,
D 1837 - 61 T, Volatility of Liquefied Petroleum (LP) Gases,
D 1838 – 61 T, Copper Strip Corrosion by Liquefied Petroleum (LP) Gases,
D 1839 – 61 T, Amyl Nitrate in Diesel Fuels, and
D 1840 – 61 T, Naphthalene Hydrocarbons in Aviation Turbine Fuels by Ultraviolet Spectrophotometry.

Editorial Change

D 286 – 58 T, Tentative Method of Test for Autogenous Ignition Temperatures of Petroleum Products:

Editorial Note.—Add a centered note between the title and Section I to read as follows:

> The committee responsible for this method urges that its use be discontinued, and plans to recommend that it be withdrawn. The improved procedure, ASTM D 2155, Test for Autoignition Temperature of Liquid Petroleum Products, should be used for the determination of autoignition temperature.

American Standards

The committee recommends the following for action by ASA Sectional Committee Z11 on Petroleum Products and Lubricants:

Reapproval of American Standards Revised in this Report:

Z11.22—1960, Drop Melting Point of Petroleum Wax, Including Petrolatum (D 127)
Z11.37—1962, Knock Characteristics of Motor Fuels at 100 Octane Number and Below by the Motor Method (D 357)
Z11.41—1961, Unsulfonated Residue of Petroleum Plant Spray Oils (D 483)
Z11.43—1960, Distillation of Petroleum Plant Spray Oils (D 447)
Z11.69—1962, Knock Characteristics of Motor Fuels at 100 Octane Number and Below by the Research Method (D 908)
Z11.101—1962, Aniline Point and Mixed Aniline Point of Petroleum Products and Hydrocarbon Solvents (D 611)
Z11.121—1962, Knock Characteristics of Motor Fuels Above 100 Octane Number by the Research Method (D 1656)

Approval as American Standard:

D 808 - 57 T, Chlorine in New and Used Petroleum Products
D 909 - 60 T, Knock Characteristics of Aviation Fuels by the Supercharge Method
D 1264 - 53 T, Water Washout Characteristics of Lubricating Greases
D 1478 - 57 T, Low-Temperature Torque of Ball-Bearing Greases
D 1548 - 59 T, Vanadium in Navy Special Fuel Oil
D 1657 - 59 T, Specific Gravity of Light Hydrocarbons by Pressure Hydrometer
D 1658 - 59 T, Carbon Number Distribution of Aromatic Compounds in Naphthas by Mass Spectrometry
D 1745 - 60 T, Calculation of Absolute Viscosity
D 1747 - 62, Refractive Index of Viscous Materials
D 2161 - 63 T, Conversion of Kinematic Viscosities to Saybolt Universal and Saybolt Furol Viscosities

American Standards Withdrawn:

Z11.46—1953, Conversion of Kinematic to Saybolt Universal Viscosity (D 446)
Z11.53—1957, Conversion of Kinematic to Saybolt Furol Viscosity (D 666)
Z11.57—1960, Sulfated Residue, Lead, Iron and Copper in Lubricating Oils (D 810)
Z11.67—1955, Saponification Number by Potentiometric Titration (D 939)

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[10]

The report has been submitted to letter ballot of the committee, which consists of 152 voting members; 106 members returned their ballots, of whom 105 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

H. M. SMITH,
Chairman.

W. T. GUNN,
Secretary.

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[10] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE D-3
ON
GASEOUS FUELS*

Committee D-3 on Gaseous Fuels held one meeting during the year on June 28, 1962, in New York, N. Y., during the ASTM Annual Meeting. Two subcommittees also met at that time, and Subcommittee V met in Atlantic City, N. J., on Oct. 9, 1962.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee D-3 requests approval by the Society at the Annual Meeting of the following recommendations. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] The existing tentatives referred to in this report appear in their present form in the 1961 Book of ASTM Standards, Part 8.

New Tentative

Tentative Method for Analysis of Liquefied Petroleum (LP) Gases by Gas Chromatography (jointly with Committee D-2 on Petroleum Products and Lubricants), as appended hereto.[2]

Revision of Standards, Immediate Adoption

(requires nine-tenths affirmative vote)

Standard Methods of Test for Specific Gravity of Gaseous Fuels (D 1070 – 52):

Section 6.—Change the Note to read as follows:

NOTE.—The sample shall be representative of the gas being sampled and shall be taken from its source without change of form or composition. For natural gases the sampling procedure specified in the Method of Sampling Natural Gas (ASTM Designation: D 1145) shall be used; and for manufactured gases the procedure specified in the Method of Sampling Manufactured Gas (ASTM Designation: D 1247) shall be used.

Method C.—After the centered heading for Method C and before Section 19 on Apparatus, change the Note to read:

NOTE.—See note immediately following Section 2. Slight changes have been made in this instrument since the original study was made. Those purchasing the new models may secure information and recommendations on their calibration and use from the manufacturer.

Section 21(h).—At the end of Note 2, add the following sentence: "A satisfactory substitute is Socony-Vacuum White Oil No. 207."

Section 22.—Change the first two sentences to read: "If calibration by means of a gas of known specific gravity is desired, standardized and certified samples may be obtained from the Institute of Gas Technology, Technology Center, Chicago 16, Ill. Pure carbon dioxide, which is commercially available at reasonable cost, may also be used."

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[2] The new tentative appears in the *1964 Book of ASTM Standards,* Part 19.

Appendix IV and Appendix V.—Add the following note at the end of these appendixes:

NOTE.—See the explanatory note preceding Section 19.

Standard Method of Test for Water Vapor Content of Gaseous Fuels by Measurement of Dew Point Temperature (D 1142 – 58):

Section 1.—Change to read as follows, deleting present Paragraphs (*b*) and (*c*) and the Note:

1. *Scope.*—This method of test covers the determination of the water vapor content of gaseous fuels by measurement of the dew-point temperature and the calculation therefrom of the water vapor content.

NOTE 1.—Some gaseous fuels contain vapors of hydrocarbons or other components that easily condense into liquid and sometimes interfere with or mask the water dew point. When this occurs, it is sometimes very helpful to supplement the apparatus in Fig. 1 with an optical attachment such as the Dewtector[a] that uniformly illuminates the dew point mirror and also magnifies the condensate on the mirror. With this attachment it is possible, in some cases, to observe separate condensation points of water vapor, hydrocarbons, and glycolamines as well as ice-points. However if the dew point of the condensable hydrocarbons is higher than the water vapor dew point, when such hydrocarbons are present in large amounts, they may flood the mirror and obscure or wash off the water dew point. Best results in distinguishing multiple component dew points are obtained when they are not too closely spaced.

NOTE 2.—Condensation of water vapor on the dew point mirror may appear as liquid water at temperatures as low as 0 F to –10 F. At lower temperatures an ice point rather than a water dew point likely will be observed. The minimum dew point of any vapor that can be observed is limited by the mechanical parts of the equipment. Mirror temperatures as low as –150 F have been measured, using liquid nitrogen as the coolant with a thermocouple attached to the mirror, instead of a thermometer well.

Section 2.—Change Paragraph (*a*) to begin "*Water Dew-Point Temperature* . . ."

Change Paragraph (*b*) to read as follows:

(*b*) *Saturated Water Vapor.*—When a gas containing water vapor is at the water dew-point temperature, it is said to be saturated at the existing pressure. Saturated water vapor, or equilibrium water vapor content, represents the water vapor concentration in a gas mixture which is in equilibrium with a liquid phase of pure water that is saturated with the gas mixture.

Section 3.—In Paragraph (*a*) line 13, delete the word "water" before the word "vapor."

In the last sentence of the last paragraph of (*b*) insert the word "water" before the words "dew points."

Section 4(a).—Change the second sentence to read as follows:

It shall not be taken at a point where isolation would permit condensate to collect or would otherwise allow a vapor content to exist that is not in equilibrium with the main stream or supply of gas, such as the sorption or desorption of vapors from the sampling line or from deposits therein.

Section 5.—Insert the word "water" before the words "dew point" throughout Paragraphs (*a*) and (*b*) including the Note.

Figure 2.—Change the caption to read "Equilibrium Water Vapor Content of Natural Gases."

Table III.—Change the title to read: "Equilibrium Water Vapor Contents of Natural Gases above the Critical Temperatures."

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[a] Several pieces of apparatus for this purpose are commercially available. Information concerning this apparatus is available from ASTM Headquarters.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee III on Determination of Calorific Value of Gaseous Fuels (A. F. Cascioli, chairman) is obtaining material based on working experiences for expansion of the present heating value range specified in the Method of Test for Calorific Value of Gases in the Natural Gas Range by Continuous Recording Calorimeter (D 1826). It is planned to circulate information among the members for their expression preparatory to the next meeting.

Subcommittee IV on Determination of Specific Gravity and Density of Gaseous Fuels (E. L. Upp, chairman) approved the revisions of Method D 1070 as noted earlier in this report. It was considered advisable to present these revisions at this time rather than to delay until an extensive evaluation of present instruments was completed and resulting additions to the method completed.

E. L. Upp, of the Tennessee Gas Pipeline Co., was recently appointed chairman of this subcommittee succeeding E. E. Stovall who resigned.

Subcommittee V on Determination of Special Constituents of Gaseous Fuels (O. W. Lusby, chairman) at its Oct. 9, 1962, meeting reviewed a preliminary report by the Institute of Gas Technology on its procedure developed for determination of hydrogen sulfide in concentrations less than one grain per 100 cu ft. As a result of the encouraging nature of the material developed, plans were formulated for cooperative testing of this method by committee members and others who had expressed their willingness to assist.

Subcommittee VI on Determination of Water Vapor Content of Gaseous Fuels (E. G. Hammerschmidt, chairman) completed the revision of Method D 1142 as noted earlier in this report. The revision acknowledges the utility of a supplemental optical attachment, recognizes its use in determining multiple dew points, and modifies the limitation of minimum dew point.

Subcommittee VII on Complete Analysis of Chemical Composition of Gaseous Fuels (D. V. Kniebes, chairman) prepared the new Tentative Method for Analysis of Liquefied Petroleum (LP) Gases by Gas Chromatography as appended to this report.[2] This method is under the joint sponsorship of Committees D-3 and D-2 on Petroleum Products and Lubricants.

Consideration is also being given to a method of determination of heating value by calculation. It is planned to distribute information prepared to interested subcommittees for their review and expression.

This report has been submitted to letter ballot of the committee, which consists of 31 members; 25 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

D. V. KNIEBES,
Chairman.

K. R. KNAPP,
Secretary.

REPORT OF COMMITTEE D-4
ON
ROAD AND PAVING MATERIALS*

Committee D-4 on Road and Paving Materials held two meetings during the year: in New York, N. Y., on June 29, 1962, and in Washington, D. C., on Jan. 30, 1963.

The committee consists of 164 members, of whom 74 are classified as producers, 54 as consumers, and 36 as general interest members.

The committee lost two members by death: B. A. Anderton and A. E. Keeley.

The committee approved a reorganization plan to become effective after the Annual Meeting in June, 1963. Subcommittees were regrouped and most were given new titles and scopes. The subcommittee membership limit of 25 was removed. An additional group of subcommittees was formed and an additional committee vice-chairman was authorized.

The officers elected in June, 1962, are as follows:

Chairman, R. E. Bollen.
First Vice-Chairman, W. H. Goetz.
Second Vice-Chairman, J. E. Gray.
Third Vice-Chairman, J. L. Wilks.
Membership Secretary, D. W. Lewis.
General Secretary, B. F. Kallas.

The committee sponsored a Symposium on Fundamental Viscosity of Bituminous Materials[2] at the 1962 Annual Meeting.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee D-4 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] All of the existing tentatives and standards referred to appear in their present form in the 1961 Book of ASTM Standards, Part 4, or the 1962 Supplement thereto.

Proposed Method to be Published as Information

Proposed Method of Test for Stripping of Bitumen-Aggregate Mixtures (Tracer Salt Method) as appended hereto.[3]

(A Report on the Statistical Analysis of Per Cent Retained Coating in Stripping of Asphalt from Aggregate by Water has been prepared by the committee and is discussed in the Appendix to this report.)[4]

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[2] Published as *ASTM STP 328*.

[3] The proposed method appears in the *1964 Book of ASTM Standards*, Part 11.

[4] See p. 353.

New Tentatives

as appended hereto.[5]

Tentative Methods of Test for:

Kinematic Viscosity of Asphalts,
Absolute Viscosity of Asphalts, and
Quantitative Extraction of Bituminous Paving Mixtures.

Tentative Specification for:

Liquid Asphalt (Slow Curing Type),
Cut-Back Asphalt (Rapid Curing Type), and
Cut-Back Asphalt (Medium Curing Type).

Revisions of Tentatives

Tentative Method of Test for Effect of Heat and Air on Asphaltic Materials (Thin-Film Oven Test) (D 1754 – 60 T):

Section 7.—Change to read as follows:

7. The following data should be used for judging the acceptability of results (95 per cent probability). The values shown are based on the concept of precision as given in the ASTM Proposed Recommended Practice for Applying Precision Data ("ASTM Methods of Test for Petroleum Products and Lubricants"):

(*a*) *Repeatability.*—Duplicate results by the same operator should be considered suspect if they differ by more than the following amount:

Percentage of retained penetration	4.0
Loss of weight:	
less than 0.4 per cent	0.04
greater than 0.4 per cent	8 per cent of the mean value

(*b*) *Reproducibility.*—The results submitted by two laboratories should be considered suspect if they differ by more than the following amount:

Percentage of retained penetration	8.0
Loss of weight:	
less than 0.4 per cent	0.16
greater than 0.4 per cent	40 per cent of the mean value

NOTE.—The precision statement is derived from the following test data:

	Repeatability and Reproducibility
Number of samples	7
Number of laboratories	26
Number of replicates per test	3

Tentative Specifications for Asphalt Cement for Use in Pavement Construction (D 946 – 62 T):

Table I.—Delete the line "Loss on heating, 325 F, 5 hr, per cent" including the test limits. Add in its place "Retained penetration after thin film oven test, per cent" with the following minimum limits:

Asphalt Penetration Grade	Retained Penetratio per cent
40–50	55+
60–70	52+
85–100	47+
120–150	42+
200–300	37+

Section 4(e).—Change the reference from Method D 6 to Method D 1754.

Tentative Methods of Test for Resistance to Deformation and Cohesion of Bituminous Mixtures by Means of Hveem Apparatus (D 1560 – 62 T):

Section 3(e).—Add to the end of Note 5 ". . . for all dense-graded bituminous mixtures containing aggregates not larger than ½ in. and within a range of 4 units for coarser graded bituminous mixtures containing up to 1-in. maximum size aggregates."

Section 11.—Change to read as follows:

11. Calculate the cohesiometer value as follows:

$$C = \frac{L}{W(0.20H + 0.044H^2)}$$

where:

C = cohesiometer value (grams per inch of width corrected to 3-in. height),
L = weight of shot, in grams,
W = diameter or width of specimen, in inches, and
H = height of specimen, in inches.

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[5] The new tentatives appear in the 1963 Supplement to *Book of ASTM Standards*, Part 4.

Tentative Method for Preparation of Test Specimens of Bituminous Mixtures by Means of California Kneading Compactor (D 1561 – 62 T):

Section 2(a).—In Note 4, add to the end of the sentence: "for all dense-graded bituminous mixtures containing aggregates not larger than ½ in. and within a range of 4 units for coarser graded bituminous mixtures containing up to 1-in. maximum size aggregates."

Section 4(b).—Add new Notes 7 and 8 at the end of this paragraph to read as follows, renumbering present Note 7 as Note 9:

NOTE 7.—When testing referee samples for the purpose of qualifying a compactor, use only material for the test specimens which will compact under the maximum foot pressure prescribed in Section 4(*b*) of Method D 1561 without visible movement, distortion, or penetration of the compactor foot.

NOTE 8.—In some instances where sandy or unstable material is involved, it may not be possible to accomplish the compaction in the kneading compactor because of undue movement of the mixture under the compactor foot. In these instances use a 40,000-lb static load (for 4-in. diameter specimen) applied by the double plunger method in which a free fitting plunger is placed on both the bottom and top of the test specimen.

Tentative Revisions of Standards

Standard Method of Test for Distillation of Cut-back Asphaltic Products (D 402 – 55):

Section 9.—Change to read as follows:

9. The following criteria should be used for judging the acceptability of results (95 per cent confidence level):

(*a*) *Repeatability.*—Duplicate results by the same operator should not differ by more than 1.0 volume per cent of the original sample, or volume per cent residue.

(*b*) *Reproducibility.*—Results by different operators in different laboratories should not differ by more than the following:

Distillation Fractions, Volume, per cent	Original Sample
up to 347 F	3.5
above 347 F	2.0
	Residue
	2.0

Section 3(b) and Fig. 3.—Change condenser tube length from "400 ± 25 mm" to "450 ± 5 mm."

Standard Methods of Testing Emulsified Asphalts (D 244 – 60):

New Test.—Add a new test for particle charge of asphalt emulsions to read as follows including the figures as shown in the accompanying Figs. 1 and 2[6]:

Scope

1. This test is made to identify cationic emulsions. Positively-charged particles are classified as cationic.

Apparatus

2. (*a*) *Charge Source,* of 12 v direct current, a milliammeter, and a variable resistor. (See Figs. 1 and 2.)

(*b*) *Plates.*—Two stainless steel plates 1 in. by 4 in., insulated from each other and rigidly held parallel ½ in. apart. (See Fig. 1.)

(*c*) *Beaker,* 150 or 250 ml.

Test Procedure

3. (*a*) Pour the emulsion to be tested into the 150- or 250-ml beaker to a height that will allow the electrodes to be immersed 1 in. in the emulsion.

(*b*) Connect the electrodes, which have been cleaned and dried, to the d-c power source and insert them into the emulsion to a depth of 1 in.

(*c*) Adjust the current to at least 8 ma with the variable resistor and start timing with a suitable timing device equipped with a second hand.

NOTE.—The 8 ma is a minimum current value—higher current levels may be specified. Current used shall be reported.

(*d*) When the current has dropped to 2 ma, or at the end of 30 min, whichever occurs first, disconnect the current source, and gently wash the electrodes in running water.

(*e*) Observe the asphalt deposit on the electrodes. A cationic emulsion will deposit an appreciable layer of asphalt on the cathode (negative electrode) while the anode (positive electrode) will be relatively clean.

Report

4. Report the test results in terms of the determined polarity (positive or negative) as defined in Section 3(*e*).

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[6] Figures 1 and 2 are not included in this report but appear in the tentative revision of Methods D 244; see 1963 Supplement to *Book of ASTM Standards,* Part 4.

Standard Methods of Testing Preformed Expansion Joint Fillers for Concrete (Nonextruding and Resilient Types) (D 545 – 49):

Section 2.—In the first sentence of Paragraph (*a*) delete "cork rubber" and "(types I, III, IV, and V.)" After "bituminous" insert the words "cork or."

In Paragraph (*b*) delete "(type II)."

Section 3.—Delete "(type II)."

Section 4.—In Paragraph (*a*) delete all references to type of filler used with a Roman numeral. Delete reference to "cork rubber." Insert "cork or" between the words "bituminous" and "fiber."

In Paragraph (*f*) delete "fiber (type V)."

Section 5(a).—Delete all references to "type" and "I, II, III, IV, and V." Delete "cork rubber." Insert "cork or" between the words "bituminous" and "fiber".

Section 6.—Delete reference to "types I and II."

Section 7(a).—Delete reference to "type II."

New Sections 10 and 11.—Add new Sections 10 and 11 to read as follows:

Water Absorption

10. A specimen 4 by 4 in. shall be cut from the joint material in such a manner that all edges are freshly cut. The specimen shall be dried in air, weighed to the nearest 0.1 g and immersed in water at a temperature of 65 to 75 F in a horizontal position with 1 in. of water over the specimen for 24 hr. The specimen shall then be removed from the water and the excess surface water removed from all sides of the specimen with a blotting paper or paper towel. The specimen shall then be quickly weighed and the percentage of absorption computed as follows:

$$\text{Absorption (by volume), per cent} = \frac{w' - w \times 100}{t \times 262}$$

where:

t = thickness of specimen before test, in inches,

w = weight of specimen before immersion, in grams, and

w' = weight of specimen after immersion, in grams.

Density

11. A specimen 4 by 4 in. shall be cut from the joint material and, after air drying, weighed to the nearest 0.1 g. The dimensions of each specimen shall be determined to the nearest 0.01 in. The weight in pounds per cubic foot shall be calculated from these determinations.

Revision of Standard and Reversion to Tentative

as appended hereto.[7]

Standard Specification for:

Emulsified Asphalt (**D 977 – 57**).

Adoption of Tentatives as Standard Without Revision

Tentative Specifications for:

Coarse Aggregates for Bituminous Paving Mixtures (**D 692 – 59 T**), and

Fine Aggregate for Bituminous Paving Mixtures (**D 1073 – 59 T**).

Tentative Methods of Test for:

Sieve or Screen Analysis of Fine and Coarse Aggregates (**C 136 – 61 T**) (Jointly with Committee C-9), and

Recovery of Asphalt from Solution by Abson Method (**D 1856 – 61 T**).

Adoption of Tentative Revision as Standard

Standard Definitions of Terms Relating to Materials for Roads and Pavements (D 8 – 55):

Adopt as standard the tentative revision issued June, 1960.

Revision of Standard, Immediate Adoption

(requires nine-tenths affirmative vote)

Standard Method of Test for Abrasion of Graded Coarse Aggregate by Use of the Deval Machine (D 289 – 55):

Section 5.—Add a new note to Paragraph (*a*) to read as follows:

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[7] The revised tentative appears in the 1963 Supplement to *Book of ASTM Standards*, Part 4.

NOTE.—For a given material, the grading used in the test may cause the percentage of wear to vary to a marked extent. This should be observed in the preparation of specification requirements.

Delete Paragraph (*c*), and reletter Paragraph (*d*) as (*c*).

Tentatives Continued Without Revision

Tentative Methods of Test for:

Coating and Stripping of Bitumen-Aggregate Mixtures (**D 1664 - 59 T**),
Concrete Joint Sealers (**D 1191 - 52 T**),
Concrete Joint Sealers, Cold-Application Type (**D 1851 - 61 T**),
Jet-Fuel-Resistant Joint Sealer for Concrete, Cold-Application Elastic Type (**D 1853 - 61 T**), and
Jet-Fuel-Resistant Concrete Joint Sealer, Hot-Poured Elastic Type (**D 1855 - 61 T**).

Tentative Specifications for:

Mineral Filler for Bituminous Paving Mixtures (**D 242 - 57 T**),
Hot-Mixed, Hot-Laid Asphalt Paving Mixtures (**D 1663 - 59 T**),
Hot-Mixed, Hot-Laid Tar Paving Mixtures (**D 1753 - 60 T**),
Crushed Stone, Crushed Slag, and Gravel for Single or Multiple Bituminous Surface Treatments (**D 1139 - 61 T**),
Mixing Plants for Hot-Mixed, Hot-Laid Bituminous Paving Mixtures (**D 995 - 61 T**),
Concrete Joint Sealer, Hot-Poured Elastic Type (**D 1190 - 52 T**),
Preformed Expansion Joint Fillers for Concrete Paving and Structural Construction (Nonextruding and Resilient Bituminous Types) (**D 1751 - 60 T**),
Preformed Expansion Joint Fillers for Concrete Paving and Structural Construction (Nonextruding and Resilient Non-Bituminous Types) (**D 1752 - 60 T**),
Concrete Joint Sealer, Cold-Application Type (**D 1850 - 61 T**),
Jet-Fuel-Resistant Concrete Joint Sealer, Cold-Application Elastic Type (**D 1852 - 61 T**), and
Jet-Fuel-Resistant Concrete Joint Sealer, Hot-Poured Elastic Type (**D 1854 - 61 T**).

Editorial Changes

Tentative Specifications for Asphalt Cement for Use in Pavement Construction (D 946 - 62 T):

Section 3(a).—Delete the words "free of water."

This report has been submitted to letter ballot of the committee, which consists of 164 members; 117 members returned their ballots, of whom 115 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

R. E. BOLLEN,
Chairman.

B. F. KALLAS,
General Secretary.

APPENDIX

REPORT ON STATISTICAL ANALYSIS OF PER CENT RETAINED COATING IN STRIPPING OF ASPHALT FROM AGGREGATE BY WATER

When aggregate is coated with asphalt and then subjected to the action of water, the asphalt coating, to a greater or lesser degree, becomes loosened or washed away.

Two methods have been developed to measure the degree of this stripping, differing principally in the final step of evaluating the degree of stripping realized by a prescribed operational procedure of coating, curing, and subjecting to the action of water. One of these methods has already been accepted as an ASTM Tentative;[1] its method of evaluating degree of stripping is by visual observation of the surface of the test specimen after soaking in water. The precision of this visual evaluation depends upon the subjective integration by the eye of numerous visible particles of aggregate, each of some varying balance of color due to partial stripping. The second method, which is now being published as information[2] uses substantially the same manipulative procedure in coating, curing, and stripping with water but depends upon an objective procedure to evaluate the actual degree of stripping realized. It eliminates the bias of the human eye by measuring stripping in terms of analyses for a tracer salt of lithium. By this procedure, the aggregate is impregnated with a very low concentration of lithium salt, which is measured by soaking the uncoated aggregate in water and determining the lithium concentration in parts per million in the stripping water by means of a flame photometer. The stripping water from another aliquot of the impregnated aggregate after coating and curing is similarly measured for lithium. The fraction of the lithium of the impregnated but uncoated aggregate aliquot which is recovered in the stripping water from the coated and cured sample measures the fraction of the coating stripped.

To provide cooperative data for a statistical comparison of appraisal of stripping by the subjective visual and by the objective lithium tracer salt procedures, five laboratories tested four combinations involving two aggregates and two cutbacks of different antistripping characteristics, by a common procedure of mixing, coating, curing, and stripping, followed by appraisal of each sample for per cent retained coating by both the visual and the lithium tracer salt techniques.

One aggregate was a No. 4 to 6 sieve size white dolomite; the other aggregate was a No. 4 to 6 sieve size mixed stone aggregate including dolomite, rhyolite, chert, granite, and river gravels. The colors of the aggregate particles ranged from white through yellows, browns, reds, to blacks with many particles of variegated color tones. The use of both uniform white and mixed color aggregate was adopted to determine if the eye distinguished asphaltic coating as well on run-of-mine aggregate as it does on uniform white aggregate.

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[1] Tentative Method of Test for Coating and Stripping of Bitumen-Aggregate Mixtures (D 1664 - 59 T); *1961 Book of ASTM Standards*, Part 4, p. 544.

[2] Proposed Method of Test for Stripping of Bitumen-Aggregate Mixtures (Tracer Salt Method); *1964 Book of ASTM Standards*, Part 11.

Large batches of each aggregate were impregnated by a very dilute water solution of lithium chloride and then dried and brought to equilibrium with 50 per cent humidity air at room temperature; this was done at a central site. Samples of the aggregate batches together with samples of the two cutbacks and samples of standardized lithium chloride solutions (0 to 20 ppm lithium) were sent to each collaborating laboratory. From these, each laboratory took aliquots for coating, curing, and stripping by the prescribed procedure. For each aggregate-cutback combination, two replicate runs were made, and were individually appraised for per cent retained coating by both the visual and the lithium salt procedures.

At each laboratory, two to four operators independently visually appraised the stripped samples. This appraisal was done before removal of stripping water for lithium analysis.

For the lithium appraisals, the stripping waters were removed from the samples after visual appraisal. Each such water was analyzed by the flame photometer and two independent readings taken for each. The base line of 100 per cent stripped (the "lithium content" of the aggregate) was determined by soaking, as in the stripping step, three samples of each impregnated but uncoated aggregate taken at the time of taking aggregate aliquots for coating, curing, and stripping. In the lithium procedure, complete recovery of stripping water from aggregate is not required; only its lithium concentration comes into the calculations. Besides making its own lithium analyses, each laboratory combined its residual stripping waters from the replicate samples of each of its several combinations and sent aliquots of these to each of the other cooperating laboratories, giving a five-way crosscheck on all lithium analyses. It was not practical, of course, to crosscheck similarly for visual appraisal. Per cent retained coating (PRC) by lithium appraisal is determined as follows:

$$\text{PRC} = 100 - 100\,(x/y)$$

where:

x = ppm lithium in the stripping water from a coated, cured, and stripped mix, and

y = ppm lithium in the stripping water from the impregnated but uncoated aggregate of that mix.

A report covering the complete statistical analysis including all data collected has been compiled. It has been distributed to members of Committee D-4 on Road and Paving Materials and a copy is on file in the permanent records of ASTM Headquarters. A limited number of copies are available upon request.

REPORT OF COMMITTEE D-5
ON
COAL AND COKE*

Committee D-5 on Coal and Coke held two meetings during the year: in New York, N. Y. on June 25 to 27, 1962, and in Montreal, Canada, on Feb. 3 to 6, 1963. In addition, meetings of the Advisory Subcommittee and Subcommittee XXVII, American Group ISO/TC-27, were held in Pittsburgh, Pa. on Sept. 24, 1962.

During the year the committee lost two members due to retirement and two resigned. Five new members were added, bringing the committee up to 70 members classified as: producers, 17; consumers, 27; and general interest members, 26. The committee consists of the Advisory and 10 subcommittees having 14 sections and task groups. Included in these groups are 36 consulting members with specialized knowledge in the sampling, testing, analysis, classification, and related activities in the evaluation of coal, coke, and fuel briquets.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee D-5 requests approval by the Society at the Annual Meeting of the following recommendations. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] All of the existing tentatives and standards referred to in this report appear in their present form in the 1961 Book of ASTM Standards, Part 8.

Tentative Revision of Standard

Standard Method of Test for Free-Swelling Index of Coal (**D 720 – 57**):

Section 6.—Change to read as follows:

6. (*a*) Report the average swelling index of a series of three buttons expressed to the nearest one-half unit. If this test is to be used for International Classification of hard coal by type, five buttons shall be made.

(*b*) Report the average swelling index of the buttons to the nearest one-half unit. Report the swelling index as zero if the residue from the test sample is noncoherent. If the residue is coherent but non-swollen, place it on a flat surface and carefully place a 500-gram weight on the button. If the button disintegrates, the swelling index shall be reported as one-half. If the button supports the weight or merely cracks into two or three hard, coherent pieces, the number shall be reported as one.

Revision of Standard and Reversion to Tentative

Standard Method of Tumbler Test for Coke (**D 294 – 50**):

Revise and revert to tentative as appended hereto.[2]

Tentatives Continued Without Revision

Tentative Methods of Test for:

Plastic Properties of Coal by the Gieseler Plastometer (**D 1812 – 60 T**), and

Fusibility of Coal Ash (**D 1857 – 61 T**).

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[2] The revised tentative appears in the *1964 Book of ASTM Standards*, Part 19.

ACTIVITIES OF SUBCOMMITTEES

Advisory Subcommittee (R. L. Coryell, chairman) has, in addition to reviewing membership matters, taken the following actions on Committee D-5 organization and policies: (1) appointed G. E. Keller and C. R. Montgomery as USA delegates to the meeting of ISO/TC-27 Working Group 7 on Sampling, in Heerlen, Netherlands, Oct. 22–24, 1962; (2) appointed two special task groups, each headed by T. Reed Scollon, vice-chairman of this committee to study long-range planning, and financing of ISO/TC-27 delegates' expenses; (3) appointed H. J. Wolthorn as chairman of Subcommittee XXI on Methods of Analysis, to replace J. B. McIlroy who had retired; (4) Appointed W. J. Montgomery as chairman of Section A on Proximate Analysis, of Subcommittee XXI; (5) reactivated Section B on Coke Sampling, of Subcommittee XXIII with D. E. Wolfson as chairman.

Subcommittee II on Nomenclature, Definitions, and Editorial, (C. H. Sawyer, chairman) has been actively engaged in reviewing new tentatives on coal and coke for consistency in terminology and definitions for various words and groups of words.

Subcommittee VII on Physical Characteristics of Coal (A. Bogot, chairman).—Work is in progress on revision of the Method of Test for Grindability of Coal, (D 409) and the Method of Sampling and Fineness of Powdered Coal (D 197). Task groups are reviewing possible need for revision of other physical tests of coal, including tests for size consist, volume, strength, and dustiness.

Subcommittee XV on Plasticity and Swelling of Coal (C. R. Montgomery, chairman) is continuing the preparation of a method for measuring pressures developed during carbonization of coal by a movable wall oven. Information for revision of the Method of Test for Free Swelling Index of Coal (D 720) is being obtained with new prototype electric furnaces. Ten such furnaces are being tested in different laboratories for interchangeability with the present standard gas burners. Comparisons in the form of interlaboratory tests are projected for the Method of Test for Plastic Properties of Coal by the Gieseler Plastometer (D 1812) using both manual and constant torque types of plastometers. As noted earlier in this report, it is recommended that this method be continued as tentative. A review is being made of other methods of measuring plasticity of coal.

Subcommittee XVIII on Classification of Coal (R. A. Glenn, chairman) is making considerable progress in revising the Specification for Classification of Coals by Rank (D 388). Contemplated revisions include changes in classification of lignitic coals, use of U. S. Bureau of Mines abbreviations, and other items.

Subcommittee XXI on Methods of Analysis (H. J. Wolthorn, chairman) is investigating methods for moisture determination including a possible referee method. Comparative tests are in progress on the direct and indirect methods. Further work appears necessary on details of the Method of Test for Fusibility of Coal Ash (D 1857) before the method can be adopted as standard. Work is in progress on improvements on the specifications for bottled gas used for the reducing atmosphere in the electric furnace, and the specifications for gold and nickel wire. Section B is preparing drafts of methods for determining chlorine and forms of sulfur in coal.

Subcommittee XXII on Physical Tests of Coke (W. L. Glowacki, chairman) has completed the revision and reversion to tentative of the Standard Method of Tumbler Test for Coke (D 294) as appended to this report.[1] A revised method of drop shatter test is being presented for committee approval. A revised method for sieve analysis of coke is being prepared by a task group. The subcommittee is working closely with Section B on Coke Sampling of Subcommittee XXIII, described below.

Subcommittee XXIII on Sampling (T. A. Miskimen, chairman).—The methods of mechanical sampling of coal, published as information, have been modified with the view of acceptance as tentative. The application of these methods to rotary car dumper samplers is being investigated. Work is in progress on revision of manual sampling procedures, now covered in Method D 492. Section B on Coke Sampling, has been reactivated with D. E. Wolfson, chairman. Investigation of coke sampling methods will be made and revisions will be considered where necessary. Section C is making tests of variance of sample preparation methods. The work of Subcommittee XXI on moisture determination is being followed closely with respect to preparation of samples.

Subcommittee XXIV on Statistics (A. A. Orning, chairman) is studying the variance of sample preparation and analysis. The analysis of data on bias and its significance are being given particular emphasis. A task group was organized to prepare recommended statistical procedures applicable to Committee D-5 work. Initial subjects to be covered relate to precision of tests, planning and analysis of interlaboratory tests, significance of bias, and analysis of variance of sampling, sample reduction, and chemical analysis.

Subcommittee XXV on Physical Testing of Fuel Briquets (J. W. Eckerd, chairman) has started work on preparation of methods for compression and drop shatter tests on fuel briquets. Task groups are now investigating the specific problems presented by coal, charcoal, and calcined briquets. Liaison with Committee D-7 on Wood is being maintained through their representative on this subcommittee.

Subcommittee XXVII, American Group ISO/TC-27 on Solid Mineral Fuels (W. H. Ode, chairman) has reviewed the usual large number of ISO/TC-27 documents furnished through the American Standards Assn., the USA member body of ISO. Comments on various documents have been prepared for distribution to other member bodies of ISO/TC-27. In addition, four Draft ISO Recommendations have been considered. Of these, the methods for volatile matter and for the crucible-swelling test (free-swelling index) were approved, and the total moisture determination disapproved. Further consideration of the Gray-King coke type test may be necessary.

The sixth meeting of Working Group 7 on Sampling, held in Heerlen, Netherlands, was attended by G. E. Keller and C. R. Montgomery, members of this subcommittee. Action by our delegates to the meeting has resulted in changes in the draft of coal sampling methods that has removed certain USA objections. It appears that the final draft is complete and acceptable except for editorial matter. The traveling expenses of one delegate were paid from the ASTM fund held for Committee D-5. This fund consists of contributions made by various segments of the USA coal industry.

A task group has been appointed by the Advisory Subcommittee to explore means to raise funds for paying traveling expenses of delegates to the ISO meetings abroad. Continued financial support by interested organizations is necessary if the USA is to participate effectively in this international effort.

This report has been submitted to letter ballot of the committee, which consists of 70 voting members; 64 members returned their ballots, of whom 63 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

R. L. CORYELL,
Chairman.

R. F. ABERNETHY,
Secretary.

REPORT OF COMMITTEE D-6

ON

PAPER AND PAPER PRODUCTS*

Committee D-6 on Paper and Paper Products held two meetings during the year: on June 28, 1962, in New York, N. Y., and on Feb. 21, 1963, in New York, N. Y.

The committee consists of 105 members of whom 88 are voting members; 34 are classified as producers, 24 as consumers, and 47 as general interest members.

Committee D-6 is working smoothly with its recent Joint TAPPI-ASTM reorganization and revival of Subcommittee V on Specifications.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1962 Annual Meeting, Committee D-6 presented to the Society through the Administrative Committee on Standards the following recommendations which were accepted on the dates indicated:

Revision of Tentative Method of Test for:

Specular Gloss of Paper at 75 Deg (D 1223 - 52 T) (Accepted March 21, 1963).

Revisions of Standards and Reversion to Tentative:

Standard Methods of Test for:

Rosin in Paper and Paperboard (D 549 - 46) (Accepted April 11, 1963),

Paraffin Content of Waxed Paper (D 590 - 42) (Accepted March 21, 1963),

Starch in Paper (D 591 - 42) (Accepted April 11, 1963),

Basis Weight of Paper and Paper Products (D 646 - 50) (Accepted April 11, 1963),

Bursting Strength of Paper (D 774 - 46) (Accepted April 11, 1963),

Effect of Heating on Folding Endurance of Paper (D 776 - 46) (Accepted April 11, 1963), and

Organic Nitrogen in Paper and Paperboard (D 982 - 50) (Accepted April 29, 1963).

All of these methods are available in their revised form as separate reprints.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee D-6 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] All of the existing tentatives and standards referred to appear in their present form in the 1961 Book of ASTM Standards, Part 6, or the 1962 Supplement.

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

New Tentatives

as appended hereto.[2]

Tentative Methods of Test for:

Arsenic in Paper and Paperboard,
Tensile Energy Absorption of Paper,
Folding Endurance of Paper by the M.I.T. Tester, and
Ink Absorption of Blotting Paper.

Revision of Tentative

Tentative Method of Test for Fiber Analysis of Paper and Paperboard (D 1030 – 55 T):

Revise as appended hereto.[3]

Revision of Standards and Reversion to Tentative

Standard Methods of Test for Folding Endurance of Paper (D 643 – 43):

Revise as appended hereto.[3]

Standard Method of Test for Absorption by Bibulous Papers of Water and Writing Ink (D 824 – 47):

Revise as appended hereto.[3]

Standard Method of Test for Opacity of Paper and Paper Products (D 589 – 44):

Revise as appended hereto.[3]

Revision of Standards, Immediate Adoption

(requires nine-tenths affirmative vote)

Standard Method of Test for Machine Direction of Paper (D 528 – 41):

Revise as appended hereto.[3]

Standard Method of Test for Ash Content of Paper and Paper Products (D 586 – 42):

Revise as appended hereto.[3]

Standard Methods of Test for Zinc and Cadmium in Paper (D 1224 – 54):

Title. — Change "Methods" to "Method."

Section 1.—Change Paragraph (*a*) to begin "This method covers..." In Paragraph (*b*) and in later sections replace "method" in titles of procedures by "procedure"; and add new Paragraphs (*c*) and (*d*) to read as follows:

(*c*) The polarographic procedure is preferred because of its greater accuracy and speed when used by competent personnel in a regular testing program. The others are satisfactory alternative procedures where a polarograph is not available.

(*d*) In case of dispute, the polarographic procedure shall be considered the standard procedure.

Sections 3 and 4.—Change Sections 3 and 4 to read as follows, changing the heading of Section 4 to read "Preparation of Specimen for Analysis":

3. Obtain the sample to be tested in accordance with the Method of Sampling Paper and Paperboard (ASTM Designation: D 585).

4. For each test unit of the sample, prepare a specimen for analysis by cutting small squares from representative portions of the test unit, and dry to constant weight at 100 to 105 C.

Footnote 5.—Change to read as follows:

[5] This polarographic procedure can be employed for zinc or cadmium or both. For example, where the procedure is used only for zinc, omit all reference to cadmium.

Sections 9, 10, and 12.—Change the word "sample" to read "specimen" in: Section 9(*a*), line 2; Section 9(*b*), in explanations of A_1, W, and A_2; Section 10(*a*), line 2; Section 10(*d*), in explanation of B; Section 12(*d*), in line 2 and in explanation of A and C.

New Section 13.—Add a new Section 13 to read as follows:

Report

13. The report shall include the following:

(*1*) Percentage of cadmium and zinc to the nearest 0.1 per cent.

(*2*) The procedure used.

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[2] The new tentatives appear in the 1963 Supplement to *Book of ASTM Standards*, Part 6.

[3] The revised tentatives and standards appear in the 1963 Supplement to *Book of ASTM Standards*, Part 6.

Withdrawal of Tentative

Tentative Method of Test for Contrast Gloss of Paper at 57.5 deg (**D 1222 – 52 T**).

Reapproval of Standards

Standard Methods of Test for:

Moisture in Paper, Paperboard, and Paperboard and Fiberboard Containers (**D 644 - 55**), with change in title to read: "Moisture Content of Paper and Paperboard by Oven Drying,"
Surface Wettability of Paper (Angle-of-Contact Method) (**D 724 – 45**),
Kerosine Number of Roofing and Flooring Felt by the Vacuum Method (**D 727 – 45**), and
Printing Ink Permeation of Paper (Castor Oil Test) (**D 780 – 46**).

Editorial Changes

Tentative Method of Test for Specular Gloss of Paper and Paperboard at 75 deg (D 1223 – 63 T):

Section 1.—Delete present Note 1 and add new Note 1 and new reference (**11**) to read as follows:

NOTE 1.—This method has recently been shown (**11**) to be also suitable for gloss measurements of most ink films on paper and paperboard. For the glossiest ink films, TAPPI Standard T 653 should be used. The fact that inks may be of different colors and diffuse reflectances has a negligible effect on measured gloss. If one had a black surface and a white surface which were otherwise identical, the white surface would measure less than one unit higher than the black surface by Method D 1223.

(**11**) R. S. Hunter, "Methods of Evaluating the Gloss of Ink Films," to be published by TAPPI.

Standard Method of Test for Internal Tearing Resistance of Paper (D 689 – 62):

Section 13.—Change Section 13 to read as follows under the heading "Precision":

13. (*a*) On the basis of studies made in accordance with the Recommended Practice for Interlaboratory Evaluation of Test Methods Used with Paper and Paper Products (ASTM Designation: D 1749), (**8,9**), the standard deviation of a test result, representing the average of ten readings, has been found to be:

(*1*) 1.5 per cent of the test result for the same material tested within the same laboratory.

(*2*) 2.5 per cent for different materials tested within the same laboratory.

(*3*) 4.5 per cent between laboratories.
The last figure may be reduced to 3.0 per cent by using a reference material for standardizing the instruments.

(*b*) In each of the above situations, two test results, each representing an average of ten readings, may be considered alike with a probability of 95 per cent when the two results agree within 2.77 times the appropriate standard deviation.

New Reference 9.—Add a new reference (**9**) to read as follows:

(**9**) Lashof, T. W., "APPA-TAPPI Reference Material Program. II. Effectiveness of a Reference Material in Reducing the Between-Laboratory Variability of TAPPI Standard T 414 m-49 for Internal Tearing Resistance of Paper," *Tappi* (in print).

American Standards

The committee recommends that the following methods, incorporating the revisions in this report, be submitted to the American Standards Assn. for adoption as American Standard:

Methods of Test for:

Machine Direction of Paper (D 528 – 63),
Ash Content of Paper and Paper Products (D 586 – 63),
Opacity of Paper (D 589 – 63 T),
Moisture Content of Paper and Paperboard by Oven Drying (D 644 – 55),
Qualitative Examination of Mineral Filler and Mineral Coating of Paper (D 686 – 59),
Internal Tearing Resistance of Paper (D 689 – 62),
Surface Wettability of Paper (Angle-of-Contact Method) (D 724 – 45),
Resistance of Paper to Passage of Air (D 726 – 58),
Kerosine Number of Roofing and Flooring Felt by the Vacuum Method (D 727 – 45),
Printing Ink Permeation of Paper (Castor Oil Test) (D 780 – 46),

Tensile Breaking Strength of Paper and Paperboard (D 828 – 60),
Titanium Dioxide in Paper (D 921 – 58),
Reducible Sulfur in Paper (D 984 – 61),
Total Chloride Content of Paper and Paper Products (D 1161 – 60),
Pinholes in Glassine and Other Greaseproof Papers (D 1221 – 58),
Zinc and Cadmium in Paper (D 1224 – 63).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Precision and Sampling (C. A. Bicking, chairman) has completed TAPPI-ASTM Proposed Standard Method for Rejection of Outlying Results which will be published in *Tappi*. A bibliography containing brief annotations of 400 items is about ready for publication as part of TAPPI-ASTM Survey of Precision Literature. Six other assignments are underway.

Subcommittee II on Paper Test Methods (T. W. Lashof, chairman) has had eight methods accepted by the Administrative Committee on Standards. Four new methods are proposed for letter ballot along with revision of one tentative, three standard methods for revision and reversion to tentative, three methods for immediate revision, one method for withdrawal, and reapproval of four methods.

Subcommittee IV on Container Board Test Methods (M. J. Clark, chairman).—Negative votes on static bending test and corrugating medium could not be resolved; new drafts are being prepared. Work is progressing on six other methods.

Subcommittee V on Specifications (L. Price, chairman) is cooperating with the Folding Paper Box Assn. in preparing specifications. The preparation of Information Sheets similar to a Buyer's Guide for Paper Procurement was discussed. A model will be available for discussion at next meeting.

Subcommittee VI, Editorial (W. R. Willets, chairman) is revising *STP 60-A* on Paper and Paperboard.

Subcommittee VII, Delegates to USA Advisory Committee on ISO/TC 6 on Paper (G. L. Adams, chairman) has discussed various ISO draft proposals and Pan-American Standards activities, and representation of a member from Committee D-6 to attend the next ISO/TC 6, SC 2 on Test Methods meeting in Paris.

This report has been submitted to letter ballot of the committee, which consists of 105 voting members; 65 members returned their ballots, of whom 55 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

H. A. BIRDSALL,
Chairman.

R. E. GREEN,
Secretary.

REPORT OF COMMITTEE D-7
ON
WOOD*

Committee D-7 on Wood and eight of its subcommittees met at the U. S. Forest Products Laboratory, Madison, Wis., on Jan. 28, 29, and 30, 1963.

The guest speaker at the dinner meeting on Jan. 29 was Henry Ford, of Madison, who gave an interesting illustrated talk on his trip around the world.

Interest in the work of the committee is indicated by the election of 24 new members, with only five resignations.

The committee noted with regret the death of two long-time members: R. P. A. Johnson, formerly chairman of Subcommittee VII on Wood Poles, and J. D. MacLean. Both were former members of the Forest Products Laboratory.

Task Group on Basic Stresses.—One of the most important tentatives of Committee D-7 is the Tentative Methods for Establishing Structural Grades of Lumber (D 245). In 1960, a special task group was appointed to revise the basic stresses that comprise a principal part of the tentative and are of significant importance to the wood industry, architects, engineers, and builders. The five-man task group has held numerous meetings, and will complete its assignment when additional significant data can be released to the task group. These data comprise the results of the mill study on Douglas fir by the Western Pine Assn. and the extensive tree density survey of western softwoods by the U. S. Forest Service with industry cooperation. This progress was reported to Subcommittee I at its Jan. 30, 1963, meeting, and the task group was instructed to continue its assignment and present a report as soon as they were able to obtain the necessary data.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee D-7 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot will be reported at the Annual Meeting.[1] All of the existing tentatives and standards referred to appear in their present form in the 1961 Book of ASTM Standards, Part 6, or the 1962 Supplement thereto.

New Tentative

Tentative Methods of Test for Structural Insulating Roof Deck, as appended hereto.[2]

Revision of Tentatives

Tentative Methods of Evaluating the Properties of Wood-Base Fiber and Particle Panel Materials (D 1037 – 60 T):

Section 1(a).—Add the following to the list of procedures:

*** Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.**

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[2] The new tentative appears in the 1963 Supplement to *Book of ASTM Standards*, Part 6.

	Sections
Compression Strength Parallel to Surface	33 to 39
Edge Thickness Swelling by the Disk Method	87 to 93
Direct Screw Withdrawal Test	102 to 108
Falling Ball Impact Test	109 to 113
Abrasion Resistance by the U.S. Navy Wear Tester	114 to 117

New Sections.—Add new Sections 1(*b*), 33 to 39, 87 to 93, and revisions to Sections 78 and 79 (now Sections 85 and 86), as appended hereto.[3]

Renumber sections and figures as required.

Tentative Definitions of Terms Relating to Wood-Base Fiber and Particle Panel Materials (D 1554 - 60 T):

Change present definition for "Flakes" to read as follows:

Flake.—A small wood particle of predetermined dimensions specifically produced as a primary function of specialized equipment of various types, with the cutting action across the direction of the grain (either radially, tangentially, or at an angle between), the action being such as to produce a particle of uniform thickness, essentially flat, and having the fiber direction essentially in the plane of the flakes, in over-all character resembling a small piece of veneer.

At the end of the present definition for "Hot-Pressing" add: "to compact and set the structure by simultaneous application of heat and pressure."

Change the present definition for "Shavings" to read as follows:

Shaving.—A small wood particle of indefinite dimensions developed incidental to certain woodworking operations involving rotary cutterheads usually turning in the direction of the grain; and because of this cutting action, producing a thin chip of varying thickness, usually feathered along at least one edge and thick at another and usually curled.

In the present definition for "Wood Wool (Excelsior)" add the word "long" before the word "curly."

Delete "term applied to" from definitions for extruded and flat-platen pressed particle boards.

Change the present definition for "Insulating Roof Deck Slab" to read as follows:

Insulating Roof Deck.—A structural insulating board product designed for use in open-beam ceiling roof construction. The product is composed of multiple layers of structural insulating board laminated together with water-resistant adhesive.

Change the last line of the description for "Particle Board Corestock" to read ". . . for use as a core for overlaying."

In the definition for "Planed-to-Caliper Hardboard" delete the word "obtain."

Add a new definition to read as follows:

Nail-Base Fiberboard Sheathing.—A specially manufactured, high-density structural insulating board product designed for use in frame construction to permit the direct application of certain exterior siding materials such as wood or cement-asbestos shingles.

Tentative Specification for Pressure Treatment of Timber Products (D 1760 - 62 T):

Section 5(*a*)(*1*).—Insert the following at the end of this paragraph: "When retentions are specified on a wood analysis basis, the methods of analysis shall be standard methods as listed in the tables. Retentions listed on this basis are minimal, and the tolerances listed above do not apply."

Section 7(*b*).—Insert at the end of the last sentence the following: "except for southern pine and ponderosa pine poles which may be resteamed once only to the limit of the specification for temperature and time. Other than this, poles of these species may be retreated twice."

Tables I to V.—Delete from Tables I, II, III, IV, and V the entire lines showing minimum durations for steam conditioning periods and vacuums.

Table I.—Change the limits for final

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[3] The new sections appear in the revised tentative; see 1963 Supplement to *Book of ASTM Standards*, Part 6.

steaming of Pacific Coast Douglas Fir, Western Hemlock, and Western Larch, to read as follows:

	General Use	Coastal Waters
Temperature, max, deg Fahr	240	240
Duration, max, hr	2	½

Under intermountain Douglas fir, reference Footnote *b* to the words "general use" and delete the column headed "Coastal Waters." Revise Footnote *b* to read: "Intermountain Douglas fir is not a suitable species for use in coastal waters."

In the column for intermountain Douglas fir, increase the permissible duration of final steaming to 1 hr, increase the minimum pressure to 75 lb per sq in., and increase the maximum permissible pressure to 150 lb per sq in.

In the column for redwood, change the incising requirement to "recommended" for general use and "required" for use in coastal waters, and change the retention requirements of creosote and creosote-coal tar solution for use in coastal waters to 20 lb per cu ft and refusal.

Add recommended retentions in jack pine, lodgepole pine, and red pine for above ground and ground contact use of acid copper chromate, ammoniacal copper arsenite, chromated copper arsenate, chromated zinc arsenate, and copperized chromated zinc chloride.

Table II.—Add recommended retention requirements by wood analysis of 12 lb per cu ft of creosote and 10.4 lb per cu ft of creosote-coal tar solution in southern pine and ponderosa pine piles for general service. (The extraction zone, number of borings, and ASTM analytical method are the same as for marine service piles.)

Add a recommended retention of 0.6 lb of pentachlorophenol per cu ft for general use in southern pine and ponderosa pine poles and 0.5 lb of pentachlorophenol per cu ft in oak, jack pine, lodgepole pine, and red pine piles. Add in the half column under coastal waters for all species "Not recommended" opposite pentachlorophenol.

Add a new column headed "Intermountain Douglas Fir" with requirements as follows:

Conditioning	Air-seasoning or heating in preservative or a combination
Steaming	Not permitted
Heating in preservative	Seasoned: 210 F and 6 hr. Green and partially seasoned: 220 F and no time limit.
Treatment	
Expansion bath temperature, max, deg Fahr	...
Final steaming:	
Temperature, max, deg Fahr	220
Duration, max, hr	3
Pressure, lb:	
min	75
max	150
Results of treatment:	
Retention, min, lb per cu ft:	
Creosote and creosote solutions:	*General Use*[a]
Creosote	10
Creosote-coal tar	12
Creosote-petroleum	12
Oil-borne preservatives:	
Pentachlorophenol	0.50
Penetration in inches or per cent of sapwood, min	½ and 85
Determination of penetration	A borer core shall be taken midway between the butt and top of each pile. Only those piles meeting the penetration requirement shall be accepted.

[a] Intermountain Douglas fir is not a suitable species for use in coastal waters.

Add recommended retentions by analysis for general use and use in coastal waters of creosote in jack pine, lodgepole pine, and red pine piles. For jack and lodgepole pine piles, retentions for general use in coastal waters are 12.8 and 19.2 lb per cu ft of creosote, respectively, the sampling zone is from 0 to 1.25 in.

from the surface, the number of borings is 20, and the ASTM analytical method is D 1860. For red pine piles, the retentions are 11.4 and 18.4 lb per cu ft of creosote for general use and coastal water use, respectively, the sampling zone is from 0 to 2.0 in., the number of borings is 20, and the ASTM analytical method is D 1860.

Table III.—Change the steam conditioning temperature and durations for southern pine and ponderosa pine poles to minimum temperature of 235 F, maximum temperature of 245, maximum duration for group A poles 17 hr and for group B poles 20 hr. (Ponderosa pine does not show any increased time for group B poles.)

Change the paragraph on conditioning for western larch and intermountain Douglas fir poles to "Air seasoning. To ensure cleanliness, the air-seasoned poles may be heated in preservative within the limits shown below." Change the maximum temperature and duration permitted for oil conditioning to 210 and 6 hr.

Add recommended retentions by wood analysis for creosote in jack pine and red pine poles with retentions for jack pines of 16 lb per cu ft and for red pine 8, 10.5, and 13.5 lb per cu ft. Add a reference to footnote *c* on the 13.5 lb retention for red pine. Assay zone for jack pine is 0.1 to 1.1 in. and for red pine 0.1 to 1.6 in. The number of borings for jack pine is 30 and for red pine, 20. (ASTM analytical method is D 1860 in both cases.)

Increase the penetration requirement for lodgepole pine poles to $\frac{3}{4}$ in. and 85 per cent of the sapwood.

Table IV.—Add the word "Required" in the line opposite "Incising" for Pacific Coast Douglas fir, western hemlock, and western larch ties and for intermountain Douglas fir. Add the words "Not required" in the same line for ashes, black, and honey locust, hickories, and black walnut ties.

Table V.—Change the heading on the column now headed Pacific Coast Douglas fir to "Douglas fir, western hemlock, and western larch" and add a footnote reference *b* after each of these species names. Add a new footnote *b* to read: "Round posts only."

Change the maximum permissible pressure for jack pine posts and for lodgepole pine posts to 150 lb per sq in.

Tentative Methods of Testing Metal Fasteners Employed in Wood Construction (D 1761 – 60 T):

Title.—Change to read: "Tentative Methods of Testing Metal Fasteners in Wood."

Section 2.—Change the first sentence to read: "Screws used for withdrawal tests shall be standard 1-in. No. 10 gage flathead common steel wood screws as described in "Bolt, Nut, and Rivet Standards." Reference a new footnote to this publication to read: "Available from the Industrial Fasteners Inst., Cleveland, Ohio."

Section 3.—Add a new Note 1 at the end of this section to read as follows, renumbering present notes accordingly:

NOTE 1.—The precision required and thus the manner of sampling and the number of tests will depend upon the specific objective of the investigation. No general criteria, therefore, can be established. However, experience has indicated that at least 10 specimens per variable are required as a minimum to give generally acceptable results. A larger number is desirable.

Section 4.—In Paragraph (*b*) change the third sentence to read: "The size of the lead hole shall be 70 per cent of the root diameter of the screw for softwoods and 90 per cent for hardwoods."

In Paragraph (*c*) change the last part of the sentence to read: ". . . and the diameter of the lead hole shall be 0.090 in. (2.3 mm) for softwoods and 0.120 in. (3.0 mm) for hardwoods."

Section 5.—Add to end of second sentence "for a period sufficiently long to bring the specimens to approximate equilibrium." Delete the third sentence. Change the fourth sentence to read "For most species of wood, exposure to these conditions will result in a moisture content of approximately 12 per cent."

Section 6(a).—Change the first sentence to read "Withdraw the screws by means of a tensile load applied to the screwhead as quickly as practical after driving, but in all cases within 1 hr."

Section 9.—Change the first sentence of Paragraph (*a*) to read: "Nails shall be bright medium diamond-point steel nails nominally 0.131 in. (3.33 mm) in diameter and $2\frac{1}{2}$ in. (63 mm) in length, with a head $\frac{9}{32}$ in. (7.14 mm) in diameter." Add at the end of this paragraph: "The actual size and details of the nails selected shall be recorded, including data on the properties of the metal."

In Note 3, delete the second sentence.

Change the first sentence of Paragraph (*b*) to read: "Screws used shall be standard $2\frac{1}{2}$-in. No. 10 gage flathead steel wood screws as described in "Bolt, Nut, and Rivet Standards." Add reference to the new footnote mentioned above in Section 2.

Delete Note 4.

Section 10.—Add a new Note 5 at the end of this section to read as follows:

NOTE 5.—The precision required and thus the manner of sampling and the number of tests will depend upon the specific objective of the investigation. No general criteria, therefore, can be established. However, experience has indicated that at least five specimens per variable are required as a minimum to give generally acceptable results. A larger number is desirable.

Section 13(a).—Change the first sentence to read: "As quickly as possible after assembly, but in all cases within 1 hr, test each specimen by tensile loading in a testing machine of suitable capacity."

Section 20(a).—Change the second sentence to read "Bearing plates should be used with the bolts, and the nuts shall be only 'finger tight.' "

Section 21.—Change items (*2*) and (*8*) to read as follows:

(*2*) General behavior of the joint under load, including load at first relaxation, the kind of failure, and similar details.

(*8*) Where desired, compressive strength parallel to grain for each component of each connector joint tested.

Figure 1.—Designate present Fig. 1 as Part A, and add a Part B as shown in the accompanying Fig. 1.[4]

Figure 2.—Designate present Fig. 2 as Part A, and add a new Part B as shown in the accompanying Fig. 2.[4] Add to the present figure caption the following: "Slightly different arrangements for measurement of deformations are shown in Parts A and B. Both are acceptable."

Adoption of Tentatives As Standard Without Revision,

Tentative Method for:

Accelerated Laboratory Test of Natural Decay Resistance of Woods (**D 2017-62 T**).

Tentative Methods of Test for:

Moisture and Creosote-Type Preservative in Wood (**D 1860-61 T**).

Tentative Specifications for:

Creosote—Coal Tar Solution (**D 391-61 T**),
Creosote—Petroleum Solution (**D 1858-61 T**), and
Petroleum for Blending with Creosote (**D 1859-61 T**).

Revision of Standards, Immediate Adoption

(requires nine-tenths affirmative vote)

Standard Methods of Testing Veneer, Plywood, and Other Glued Veneer Construction (D 805-52):

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[4] Figures 1 to 3 are not included in this report but appear in the revised methods; see 1963 Supplement to *Book of ASTM Standards*, Part 6.

Section 70.—Change to read as follows:

70. The test specimens shall be sawed in such a manner that, when the specimens are loaded, the lathe checks in the center ply of 50 per cent of the test specimens will tend to open, while in the remaining 50 per cent, the lathe checks in the center ply will tend to close. A method of accomplishing this requirement in one size of panel is illustrated in Fig. 19. The notches shall be cut to proper width, depth, and location, using a hollow-ground grooving saw or any other method that will give satisfactory results. The individual specimens may then be cut from the panel. The width of the specimen and the distance between notches shall be measured to the nearest 0.01 in. to determine their shear area.

New Figure.—Add a new Fig. 19 as shown in the accompanying Fig. 3.[4] Renumber present Figs. 19 and 20 as Figs. 20 and 21.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Specifications for Timber (L. W. Wood, chairman) has appointed a new task group to study the relation of new methods of machine grading stress-rated lumber to the existing recommended practice for load-sharing lumber members. It is recognized that the new recommended practice is important as a means of obtaining design stresses without the use of basic stresses.

Progress of the task group on basic stresses is reported on in the beginning of this report because of its wide importance and significance.

Subcommittee V on Methods of Preservative Treatment of Timber (C. W. Best, chairman) has recommended revisions of Specification D 1760, which mostly follow revisions of the American Wood Preservers Assn., but some others differ significantly in substance. These actions form a departure from previous practice in which an attempt was made to coordinate closely ASTM and AWPA specifications and were taken after a very thorough debate. This permits immediate action on some needed changes, and it is expected that the AWPA will eventually also agree on the same revisions.

Subcommittee VI on Timber Preservatives (D. L. Davies, chairman) has recommended adoption as standard of Specifications D 391, D 1858, D 1859, and Methods D 1860.

Subcommittee VII on Wood Poles (Ralph Bescher, chairman) has concluded its work on the strength of poles for distribution and transmission lines, and discussion has centered around the use of poles for other purposes. It is recognized that while the use of a pole in a telephone or utility line is essentially a cantilever, many poles are used as building columns and as such are not a simple cantilever beam. The subcommittee will attempt to set up strength ratings for poles of different species, based on ASTM Methods D 245 and employing calculations to convert the values from square to round.

The subcommittee will also review the ASTM pole data for strength developed in the top. This would calculate the strength that is actually developed, but not the breaking strength. Included also will be a review of the Bell Telephone Laboratory data on the breaking of poles and breaking of pole tops and of the data being collected by the Forest Products Laboratory on the effect of height on the density of wood in a tree.

Subcommittee IX on Methods of Testing (A. D. Freas, chairman). A number of changes in the Methods of Testing Metal Fasteners (D 1761) are being recommended. Inadvertently, these changes were omitted from the ballot of the main committee in the spring of 1962. In addition, a task force will be appointed to consider recommendations to the subcommittee for additional methods.

A task force charged with preparation of definitions of terms related to fastenings has reported agreement on a section related to terms for nails. The subcommittee will ballot on these definitions, and, if the vote is favorable, submit a recommendation to the full committee for ballot.

The Methods of Static Tests of Timbers in Structural Sizes (D 198) will be brought up to date and consideration given to the possibility of adding other methods. A task force will report to the subcommittee at the next meeting.

A task force has been appointed to review the literature on methods of test for specific gravity and submit recommendations to the subcommittee as to whether or not a method on this subject should be developed.

A task force has been appointed to develop recommendations relating to simulated service tests of wood and wood-base flooring.

Subcommittee XI on Moisture Content of Timber (R. C. Rietz, chairman) is giving consideration to the factors that influence the accuracy and reliability of electrical meters. The procedures for their use as now written should be amplified as experiences with their use expand. A suggested change in the distillation method has been deferred pending a review by some of the subcommittee members. A suggestion to include the Karl Fischer method for moisture content determination in the Methods has also been deferred as its use seems to be very limited.

The role of electrical moisture meters in the processing of wood was presented at a meeting of the committee.

Three recommendations have been suggested for subcommittee consideration: (1) develop a form for inclusion in the standard for tabulating and reporting data when the oven-drying method is used; (2) develop a standard for determining the shrinkage characteristics of wood, involving a technique or techniques and methods of drying or conditioning; and (3) Develop a standard for determining the electrical properties of wood for use in calibrating electrical moisture meters.

Subcommittee XIII on Durability and Exposure (T. C. Scheffer, chairman) has reviewed the three existing Methods: soil block (D 1413), $\frac{3}{4}$-in. stakes (D 1758), and natural resistance tests (D 2017). A four-member task force was appointed for the purpose of drafting proposals for revision of Method D 1413.

The subcommittee is recommending that Method D 2017 be adopted as standard.

Three tests, described as the "post test," "**marine-borer test,**" and the "particle and fiberboard test," are under consideration.

The marine-borer test has presented particular difficulties because of need for clearer definition of objectives and because of a relatively limited background of observations for guidance to preferable materials and procedures. It is probable that a preliminary draft of a marine-borer test will be ready for consideration at the next meeting.

The durability test for particle and fiberboard is the most recent one being studied. It appears that with a few modifications a test of particle and fiberboard can be essentially the same as that used in testing methods of decay resistance. Specific proposals relative to both this and the marine-borer test probably will be available at the next meeting of the committee.

Subcommittee XV on Wood-Base Fiber and Particle Panel Materials (W. C. Lewis, chairman).—One of the principal accomplishments during the year has been completion of the new "Tentative

Methods of Test for Structural Insulating Roof Deck, as appended to this report.[1] Revisions were recommended for Methods D 1037 and Definitions D 1554.

Other assignments underway are: surface smoothness test procedures, hardboard specification, insulating roof deck specification, and particle board specification requirements.

Subcommittee XVI on Wood Flour (H. W. Shader, chairman) was established several years ago to develop specifications for wood flour, but because of a change in assignment of the chairman, the work has not been activated. An organization of the subcommittee is now in prospect, and it is hoped that active work will get underway during the coming year.

This report has been submitted to letter ballot of the committee, which consists of 132 members; 97 members returned their ballots, of whom 91 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

L. J. MARKWARDT,
Chairman.

W. A. OLIVER,
Secretary.

REPORT OF COMMITTEE D-8

ON

BITUMINOUS AND OTHER ORGANIC MATERIALS FOR ROOFING, WATERPROOFING, AND RELATED BUILDING OR INDUSTRIAL USES*

Committee D-8 on Bituminous and Other Organic Materials for Roofing, Waterproofing, and Related Building or Industrial Uses held two meetings during the year: in New York, N. Y.., on June 26, 1962, and in Washington, D. C. on Feb. 1, 1963.

The membership consists of 100 voting members, of whom 59 are classified as producers, 20 as consumers, and 21 as general interest members.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee D-8 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] The existing tentatives referred to appear in their present form in the 1961 Book of ASTM Standards, Part 4, or the 1962 Supplement thereto.

New Tentative

Tentative Specification for Asphalt-Impregnated Glass Fiber Mat (Felt), as appended hereto.[2]

Adoption of Tentatives as Standard Without Revision

Tentative Methods of Test for:

Sieve Analysis of Granular Mineral Surfacing for Asphalt Roofing and Shingles (**D 451 - 61 T**),
Sieve Analysis of Nongranular Mineral Surfacing for Asphalt Roofing and Shingles (**D 452 - 61 T**),
Moisture in Mineral Aggregate for Use on Built-Up Roofs (**D 1864 - 61 T**), and
Hardness of Mineral Aggregate for Use on Built-Up Roofs (**D 1865 - 61 T**).

Tentative Specifications for:

Woven Glass Fabrics Treated with Bituminous Substances for Use in Waterproofing (**D 1668 - 61 T**), and
Mineral Aggregate for Use on Built-Up Roofs (**D 1863 - 61 T**).

Tentatives Continued Without Revision

Tentative Methods of Testing:

Asphalt Roll Roofing, Cap Sheets, and Shingles (**D 228 - 57 T**), and
Asphalt-Base Emulsions for Use as Protective Coatings for Built-Up Roofs (**D 1167 - 58 T**).

Tentative Specifications for:

Homogeneous Bituminized Fiber Drain and Sewer Pipe (**D 1861 - 61 T**), and
Laminated-Wall Bituminized Fiber Drain and Sewer Pipe (**D 1862 - 61 T**).

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* Sixty-sixth Annual Meeting of the Society, June 23-28, 1963.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[2] The new tentative appears in the 1963 Supplement to *Book of ASTM Standards*, Part 4.

Tentative Method of Test for:

Translucency of Mineral Aggregate for Use on Built-Up Roofs (**D 1886 - 61 T**)

Editorial Change

Tentative Specification for Asphalt Shingles Surfaced with Mineral Granules (**D 225 - 62 T**):

Section 1(b).—Change the maximum exposure to weather of hexagonal tab strips from "$5\frac{2}{3}$" to read "$4\frac{2}{3}$" in.

This report has been submitted to letter ballot of the committee, which consists of 100 members; 61 members returned their ballots, of whom 54 have voted affirmatively, and 0 negatively.

Respectfully submitted on behalf of the committee,

W. C. CULLEN,
Chairman.

H. B. KEENE,
Secretary.

REPORT OF COMMITTEE D-9
ON
ELECTRICAL INSULATING MATERIALS*

Committee D-9 on Electrical Insulating Materials held three meetings during the year: in New York, N. Y., on June 24 to 26, 1962, during the Annual Meeting of the Society; in Boston, Mass., on Oct. 24 to 26, 1962; and in Cleveland, Ohio, on Feb. 11 to 13, 1963.

At the time of the Cleveland meeting, the total membership comprised 358, of whom 222 were classified as official voting representatives, made up of 94 producers, 81 consumers, and 47 general interest members.

No major organizational changes have been made during the year.

Committee D-9 has continued its cooperation with other groups concerned with electrical insulation, including the International Electrotechnical Commission (IEC), the International Organization for Standardization (ISO), the Institute of Electrical and Electronic Engineers (IEEE), the Electronic Industries Association (EIA), and the National Electrical Manufacturers Association (NEMA). The committee has continued its participation in the technical programs of the IEEE-NEMA Electrical Insulation Conference—Materials and Applications, and is planning two sessions, one on "Dielectric Loss Measurements" and one on "Surface and Discharge Failure Mechanisms," at the fifth meeting of this conference, to be held in Chicago, Ill., Sept. 17 to 19, 1963.

At the Cleveland meeting on Feb. 13, 1963, Gerald H. Mains was elected to Honorary Membership in Committee D-9.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1962 Annual Meeting, Committee D-9 presented to the Society through the Administrative Committee on Standards the following recommendations which were accepted on the dates indicated:

New Tentative Methods of Test for:

Dust and Fog Tracking and Erosion Resistance of Electrical Insulating Materials (D 2132 - 62 T) (Accepted Oct. 17, 1962),

Dielectric Constant (Permittivity) and Dissipation Factor of Solid Ceramic Dielectrics at Frequencies to 10 Mc and Temperatures to 500 C (D 2149 - 63 T) (Accepted April 11, 1963), and

Bondable Silicone Rubber Tapes Used for Electrical Insulation (D 2148 - 63 T) (Accepted April 11, 1963).

New Tentative Specifications for:

Natural Muscovite Mica Splittings (D 2131 - 62 T) (Accepted Oct. 17, 1962).

Revision of Tentative Methods of Testing:

Rigid Sheet and Plate Materials Used for Electrical Insulation (D 229 - 63 T) (Accepted Sept. 10, 1963).

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* Sixty-sixth Annual Meeting of the Society, June 23-28, 1963.

Revision of Tentative Specifications for:

Fully Cured Silicone Rubber-Coated Glass Fabric and Tapes for Electrical Insulation (D 1931 - 62 T) (Accepted Oct. 17, 1962).

Withdrawal of Tentative Methods of Testing:

Flexible Vinyl Plastic Coated Sleeving Used for Electrical Insulation (D 1678- 59 T) (Accepted Oct. 17, 1962).

Withdrawal of Standard Specifications for:

Low and Medium Voltage Pin-Type Lime-Glass Insulators (D 730 - 49) (Accepted April 29, 1963).

The following items accepted on Oct. 17, 1962, appear in the 1962 Supplement to the Book of ASTM Standards, Part 11: D 2131 and D 2132 - 62 T. The other items are available as separate reprints.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee D-9 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] All of the existing standards and tentatives referred to appear in their present form in the 1961 Book of ASTM Standards, Part 11.

Adoption of Tentative as Standard With Revision

Tentative Methods of Testing Vitrified Ceramic Materials for Electrical Applications (**D 116 - 61 T**):

Section 5.—After the sentence beginning "For tests under Method C 369 . . . ," insert the following: "To conform to other accepted practice (MIL-I-10), specimens may also be 0.50 in. or 1.125 in., as well as 0.75 in., in diameter, whichever diameter is comparable to that of the finished product." (This revision of the modulus of rupture test specimen dimensions removes the final objection to withdrawal of the Standard Methods of Testing Steatite Used for Electrical Insulation (D 667 - 44) which is recommended elsewhere in this report.)

New Sections 27 to 31.—Under a centered heading "*Method D. Dye Penetration,*" add new Sections 27 to 31 to read as follows, renumbering present Sections 27 to 31 as Sections 32 to 36:

Apparatus

27. The apparatus shall consist of a suitable open air chamber of such dimensions as to accommodate the test specimens when immersed in the dye solution.

Reagent

28. *Fuchsine Solution.*—The solution of Section 23 is suitable.

Test Specimens

29. The specimens of Section 24 are suitable.

Procedure

30. (*a*) Place the test specimens in the chamber and immerse completely in the fuchsine solution.

(*b*) Permit the specimens to remain immersed for 5 min or longer, and then remove, dry thoroughly, and break as soon as possible for visual examination.

(*c*) Porosity is indicated by penetration of the dye into the ceramic body to an extent visible with the unaided eye. Disregard any penetration into small fissures formed in preparing the test specimens.

Report

31. The report shall include a statement of the observations recorded in accordance with the examination prescribed in Paragraph 30(*c*).

Section 13.—In Paragraph (*b*), line 5, add "on new specimens" before "at each level."

Change Paragraph (*c*) to read as follows:

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[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

(*c*) Immerse the specimens in fuchsine solution (Section 23) for 10 min, remove, and dry thoroughly. Examine for fracture, crazing, etc., under a bright light in accordance with Paragraph 25(*a*). If so specified, determine the modulus of rupture after thermal shock by the procedure specified in Section 5.

Adoption of Tentatives as Standard Without Revision

Tentative Methods of Test for:

Absorbed Gamma Radiation Dose in the Fricke Dosimeter (**D 1671 – 59 T**) (Jointly with Committee D-20).

Tentative Specifications for:

Vulcanized Fibre Sheets, Rods, and Tubes Used for Electrical Insulation (**D 710 – 61 T**).

Adoption of Tentative as Standard With Editorial Change

Tentative Methods of Testing Vulcanized Fibre Used for Electrical Insulation (**D 619 – 59 T**):

Section 5.—Change to read as follows:

5. (*a*) *Test Specimen.*—The test specimen shall consist of 2 to 5 g of finely divided particles, such as millings or filings, of the material.

(*b*) *Procedure.*—Dry the test specimen for 2 hr at 105 to 110 C and weigh. Then ignite the specimen to constant weight in a crucible and reweigh. Calculate the percentage of ash, based on the weight of the dried specimen.

Revision of Standards, Immediate Adoption

(Requires nine-tenths affirmative vote)

Standard Methods of Testing Pasted Mica Used in Electrical Insulation (D 352 – 59):

Revise by replacing Sections 1 to 21 with new Sections 1 to 11, as appended hereto.[2]

Standard Methods of Testing Polymerizable Embedding Compounds Used for Electrical Insulation (D 1674 – 61):

Revise by addition of test methods for dielectric constant and dissipation factor and for dielectric strength, as appended hereto.[2]

Withdrawal of Standards

The committee recommends withdrawal of the following standards because of lack of use or because their essential subject matter is being, or already has been, incorporated into other methods:

Standard Methods of Test for Product Uniformity of Phenolic Laminated Sheets (**D 634 – 44**), and
Standard Methods of Testing Steatite Used for Electrical Insulation (**D 667 – 44**).

Tentatives Continued Without Revision

The committee recommends the continuation of all other tentatives under its jurisdiction not otherwise mentioned, because the work currently in progress on their revision or because of insufficient experience in their use to justify advancement to standard.

Editorial Change

Standard Method of Test for High-Voltage, Low-Current Arc Resistance of Solid Electrical Insulating Materials (**D 495 – 61**):

Section 4(c).—Add a new Note 6 to read as follows, renumbering subsequent notes accordingly:

NOTE 6.—It is usually not possible to observe supply voltage fluctuations during the first three steps of the test specified in Table I because of cyclical impedance-voltage drops in the primary circuit which includes the autotransformer. A means shall be provided to maintain constant primary voltage after the variable autotransformer is adjusted. One of many commercially available line voltage stabilizers will provide a suitable means.

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[2] The new sections are not included in this report but appear in the revised standards; see *1964 Book of ASTM Standards*, Part 29.

Section 14.—Change Paragraphs (*a*), (*b*), and (*c*) to read as follows, and designate present Paragraph (*b*) including the Note as Paragraph (*d*):

14. (*a*) Place the specimen in a suitable holder to locate the test surface as specified in Sections 9 and 12 and insert under the tungsten rod electrodes. Alternatively place the specimen in a suitable holder and apply alignment jig and stainless steel strip electrodes as in Fig. 6; insert under the tungsten rods and remove the alignment jig as illustrated in Fig. 5.

(*b*) Lower the draft shield, close the line switch, and adjust *Ta* if necessary to obtain the voltage specified in Section 11(*b*).

(*c*) Begin the test sequence specified in Table I and simultaneously start the interval timer. At the end of each minute increase the severity in steps as shown in Table I until a failure occurs as defined in Section 3. Immediately interrupt the arc current and stop the interval timer. Record the seconds to failure.

Section 15(*a*).—Add the following Sentence: "Application of a continuous 40-ma arc, without a specimen in place, for approximately 1 min often proves to be the most effective technique of cleaning the electrodes."

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Insulating Varnishes, Paints, and Lacquers (J. S. Hurley, Jr., chairman) sponsored a Symposium on Thermal Stability and Classification of Insulating Varnishes at the Cleveland meeting on Feb. 12, 1963. A new section on conformal coatings has been approved by the D-9 Executive Committee and will be organized in the near future. The section on high-temperature varnishes is being reactivated. Gel time procedures are under study by a task force. Discrepancies in the Method of Test for Thermal Endurance of Flexible Varnishes (D 1932) are being corrected. A proposal to adopt the Brookfield and eliminate the MacMichael viscometer for use in the Methods of Test for Insulating Varnishes (D 115) is being considered.

Subcommittee II (Joint D-9/D-20) on Effects of Nuclear and High Energy Radiation (D. J. Metz, chairman) was responsible for the recommendation to adopt Method D 1671 as standard. The subcommittee has prepared a proposed method of calculation of neutron dose to organic materials and application of threshold-foil measurements. Work on the oxalic acid dosimeter for gamma radiation is in progress. An effort is being made to determine the extent to which the Recommended Practice for Exposure to Polymeric Materials to High Energy Radiation (D 1672) is being used. There is also considerable concern over the problems involved in making dielectric measurements on materials while in the presence of a radiation field. To date these problems have not been satisfactorily resolved. A proposal has been made to extend the scope of Subcommittee II to include "space environments." The subcommittee continues to maintain close liaison with Subcommittee II of Committee E-10 on Radioisotopes and Radiation Effects.

Subcommittee III on Plates, Tubes, Rods, and Molded Materials (Kenneth Wechsler, chairman) was responsible for the revisions of Methods D 619 and D 229 as accepted by the Administrative Committee on Standards; also for the recommendation to adopt Method D 619 and Specification D 710 as standard and for the recommendation to withdraw Method D 634. The development of methods for performing dielectric breakdown tests on sheets and other types of specimens in air rather than in an oil medium is under consideration. The subcommittee has prepared a Test Procedure for Thermal Evaluation of Rigid Electrical Insulating Materials and has submitted it for committee letter ballot for approval.

Subcommittee V on Ceramic Products (W. W. Perkins, chairman) was responsible for the new Tentative Method for Dielectric Constant and Dissipation Factor of Solid Ceramic Dielectrics at Frequencies to 10 Mc and Temperatures to 500 C and for the withdrawal of Specification D 730, as accepted by the Administrative Committee on Standards. The subcommittee has also recommended revision of Methods D 116 and its adoption as standard, and has recommended the withdrawal of Methods D 667. The subcommittee is working on test methods for dielectric constant and loss measurements on ceramics at microwave frequencies.

Subcommittee VI on Solid Filling, Treating, Encapsulating, and Embedding Compounds (Thomas Hazen, chairman) was responsible for the revision of Methods D 1674 by the addition of dielectric constant and dissipation factor test methods and a method for dielectric strength, as appended to this report.[2] Work is in progress on a proposed method for the effect of high humidity on polymerizable embedding materials. Liaison with a group in Committee F-1 on Materials for Electron Tubes and Semiconductor Devices is being maintained because of that group's interest in encapsulating and embedding materials for use in microelectronics.

Subcommittee VII on Flexible Sheet, Tape, and Tubing (F. P. West, chairman) was responsible for the new Tentative Method of Test for Adhesion of Bondable Silicone Rubber Tapes, for the revision of D 1931, and for the withdrawal of Method D 1678, as accepted by the Standards Committee. Work on revisions of methods and specifications for vinyl tubing, proposed methods for bond strength of fusible silicone rubber tapes, and on proposed methods for polymeric films, is in progress. New sections on harnessing tapes and base fabrics have been organized. Interlaboratory evaluations for thermal endurance on four Class F coated materials are in progress. Methods of testing automatic-closure and heat-shrinkable tubing are being developed.

Subcommittee VIII on Insulating Papers (C. F. Ackerman, chairman) has developed three new test methods for a revision of the Methods of Sampling and Testing Untreated Paper (D 202): one on sampling, one for holes and felt hair inclusions, and one for dimensions of rolls, sheets, and cores. A study is under way to determine whether currently used methods for paper testing are suitable for testing synthetic fiber papers. Task groups are engaged in revisions of methods D 202 tests for dielectric strength, extract of paper, and chlorides in paper; also in the preparation of specifications for paper used in electrolytic capacitors, in development of life tests for capacitors, and in devising methods for testing impregnated papers.

Subcommittee IX on Mica Products (K. G. Coutlee, chairman) was responsible for preparation of the new Tentative Specifications for Natural Muscovite Mica Splittings (D 2131) accepted by the Administrative Committee on Standards; also for the revision of Methods D 352, appended to this report.[1] The subcommittee has continued its activities relating to the work of ISO/TC 56 on Mica.

Subcommittee X on Magnet Wire Insulation (K. N. Mathes, chairman) has prepared a proposed new Tentative Test Method for Determining the Relative Thermal Endurance and a Temperature Value of Film-Insulated Round Magnet

Wire and the Combination of Magnet Wire with Insulating Varnish in Air, which has been submitted for committee letter ballot. The subcommittee is at work on several test methods for magnet wire insulation.

Subcommittee XI on Nomenclature, Significance, and Statistics (E. B. Snyder, chairman) has prepared a revision of the Definitions of Electrical Terms (D 1711) which has been accepted for committee letter ballot. Proposed Committee F-1 definitions relating to electronic circuit packaging have been reviewed and comments submitted to ASTM Headquarters. An extension of the definition of "conditioning" to include factors other than temperature and humidity is being contemplated. The subcommittee has continued to function in its accepted rôle as a review board for all new and revised methods and specifications to ensure conformity with correct ASTM usage.

Subcommittee XII on Electrical Tests (H. S. Endicott, chairman) is working actively on revisions of Methods D 149, D 150, and D 257. Two tracking methods have been proposed for letter ballot of the subcommittee, and an interlaboratory test on a medium power arc resistance method is under way. A proposed corona resistance method has been prepared for subcommittee ballot, and a revision of Method D 1868 is in progress. The section on electrostatic phenomena is being reactivated because of an apparent revival of interest in the subject.

Subcommittee XIII on Composite Insulating Materials (L. J. Timm, chairman) is working on the development of testing procedures for volume resistance, dielectric breakdown, and tear resistance of flexible composite insulation. A task group is engaged in preparing a tensile strength test method for these materials.

Subcommittee XIV (Joint D-9/D-20) on Conditioning (G. M. Armstrong, chairman) has been placed, at the request of the chairman, in an inactive status pending new assignments.

Subcommittee XV on Research and Technical Papers (C. L. Craig, chairman) has continued to keep the committee informed about the joint NEMA-EIA-ASTM research project on insulation resistance and about the NEMA-EIA-IPC-ASTM study of shear and punching qualities of laminates, both of which are in progress at the University of Delaware. Two ASTM-sponsored sessions, one on Dielectric Loss Measurements and one on Surface and Discharge Failure Mechanisms are being organized for the IEEE-NEMA Electrical Insulation Conference to be held in Chicago, Ill., September 17–19, 1963. The subcommittee arranged an informal session on Liquid Immersion Methods for Dielectric Measurements at the October, 1962, D-9 meeting in Boston, with K. G. Coutlee as keynote speaker. A Symposium on Thermal Stability and Classification of Insulating Varnishes was held at the Committee D-9 meeting on Feb. 12, 1963, with W. E. Harvey as chairman. A future Subcommittee VIII-sponsored symposium on Thermally Upgraded Electrical Insulating Papers is being planned.

This report has been submitted to letter ballot of the committee, which consists of 358 members; 212 members returned their ballots, of whom 169 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

ARNOLD H. SCOTT,
Chairman.

THOMAS HAZEN,
Recording Secretary.

REPORT OF COMMITTEE D-10

ON

PACKAGING*

Committee D-10 on Packaging held one meeting during the year in Columbus, Ohio, at the Battelle Memorial Inst. on Oct. 4, 1962. The subcommittees also met in Madison, Wis. on April 24, 25, and 26, 1963. J. W. Goff was appointed chairman of Committee D-10 by the Advisory Committee to fill the vacancy resulting from the resignation of K. W. Kruger, former chairman. K. Q. Kellicutt was appointed vice-chairman.

The roster of Committee D-10 is under review by H. E. Lockhart, membership secretary, and an accurate breakdown of the membership is being prepared.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1962 Annual Meeting, Committee D-10 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Standard and Reversion to Tentative

Standard Methods of Test for:

Water Vapor Permeability of Packages (**D 895 – 51**), and

Incline Impact Test for Shipping Containers (**D 880 – 50**).

The recommendation for Method D 895 was accepted by the Standards Committee on Oct. 31, 1962, and that for Method D 880 on March 26, 1963. The revised methods are available as separate reprints.

The committee recommends the continuation without revision of all tentatives under its jurisdiction. The majority of the present tentatives are under review or are scheduled for review by the governing subcommittee in the near future.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Methods Appraisal (C. R. Gustafson, chairman) discussed and approved the formation of a group to study loading and bracing practices.

Subcommittee III on Moisture and Water Vapor Resistance (H. A. Bergstrom, chairman) has completed editorial revision of the Method for Water Vapor Permeability of Packages by Cycle Method (D 1251) and for Water Vapor Permeability of Shipping Containers by Cycle Method (D 1276). Both will be submitted to Committee D-10 for letter ballot approval.

Subcommittee V on Revolving Drum Test (M. J. Clark, chairman), having completed its work on Method D 782, has been placed on inactive status.

Subcommittee VI on Interior Packing (C. E. Swanson, chairman) continues its study of compatibility of cushioning materials with lading; vibration characteristics of cushioning materials; and review of Tentative Methods D 1372 and D 1596. The proposed method of test for creep properties of package cushioning materials is being balloted upon by Committee D-10.

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

Subcommittee VII on Stacking Test (H. G. Nelson, chairman) continues its development of dead load stacking data required for the preparation of a test procedure.

Subcommittee VIII on Vibration Test (S. G. Guins, chairman) continues refinement of Method D 999 with emphasis on compatibility with proposed vibration test procedures for Federal Standard 101A.

Subcommittee IX on Incline Impact Test (R. N. Maxson, chairman) has revised Method D 880 and continues its study of variables to determine whether further refinement by definition and measurement of these variables is practicable.

Subcommittee X on Exterior Packing (H. W. Maas, chairman) continues active study of test methods for container fasteners and tapes.

Subcommittee XI on Loading, Bracing, and Related Handling (chairman to be appointed) is in process of organization. The present task group on inflatable dunnage will come under the jurisdiction of this new subcommittee.

This report has been submitted to letter ballot of the committee, which consists of 123 members; 93 members returned ballots, of whom 89 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. W. GOFF,
Chairman.

R. F. UNCLES,
Secretary.

REPORT OF COMMITTEE D-11

ON

RUBBER AND RUBBER-LIKE MATERIALS*

Committee D-11 on Rubber and Rubber-Like Materials and its subcommittees met twice during the year: in New York, N. Y., on June 29, 1962, and in Montreal, Canada, on Feb. 8, 1963.

The fiftieth Anniversary of Committee D-11 was observed at the New York City meeting. The committee was appointed in December, 1911, and the first meeting for the organization and selection of subcommittees was held in New York at the Chemists Club on Feb. 24, 1912. A special Golden Anniversary Dinner was held at the Waldorf Astoria Hotel on June 28, 1962. A. E. Juve, chairman of the Anniversary Committee, introduced O. M. Hayden, past-chairman of the committee, who reviewed briefly the highlights of Committee D-11 history; M. N. Clair, then president of ASTM; T. A. Marshall, Jr., Executive Secretary of ASTM; and G. C. Maassen, the newly elected chairman of Committee D-11. Special certificates and appropriate gifts were presented by Mr. Maassen to Simon Collier, H. G. Bimmerman, and J. J. Allen, the retiring officers of the committee. The main speaker of the evening was Tennyson Guyer, Cooper Tire and Rubber Co., also a member of the Ohio Legislature. His topic was "Don't Be Confused."

Committee D-11, at the Montreal meeting, approved the contribution of $500 to the ASTM Building Fund.

Also, at the meeting in Montreal, the committee bestowed honorary memberships on Simon Collier, J. J. Allen, and Harlan Depew. A similar honor was accorded H. G. Bimmerman at the Annual Meeting in New York.

The committee mourned the passing of W. L. Sturtevant, an honorary member; on Dec. 7, 1962.

On Dec. 1, 1962, J. F. Kerscher, Goodyear Tire and Rubber Co., replaced J. J. Allen as secretary of Committee D-11. Mr. Allen will be retiring from Firestone Tire and Rubber Co. in June, 1963.

R. T. Zimmerman, R. T. Vanderbilt Co., has been appointed chairman of Subcommittee XV on Life Tests for Rubber Products, replacing G. C. Maassen, now chairman of Committee D-11. R. F. Jones, Firestone Tire and Rubber Co., is the new secretary of this subcommittee. M. F. Torrence, E. I. duPont de Nemours & Co., Inc., was appointed secretary of Subcommittee VIII on Nomenclature and Definitions.

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1962 Annual Meeting, Committee D-11 presented to the Society through the Administrative Committee on Standards, the following recommendations which were accepted on the dates indicated, and appear in the 1962 Supplement to Book of ASTM Standards, Part 11:

New Tentatives:

Specifications and Methods of Tests for Rubberized Curled Hair (D 2128 - 62 T) (Accepted September 11, 1962),

Classification for Hard Rubber Materials (D 2135 - 62 T) (Accepted December 17, 1962),

Method for Low Temperature Bend Test for Fabrics Coated with Rubber and Rubber-Like Materials (D 2136 - 62 T) (Accepted December 17, 1962),

Method for Low Temperature Impact Test for Fabrics Coated with Rubber and Rubber-Like Materials (D 2137 - 62 T) (Accepted December 17, 1962),

Method of Test for Static Adhesion of Textile Cord to Rubber (H Pull Test) (D 2138 - 62 T) (Accepted December 17, 1962), and

Method of Test for Stress Relaxation of Gasket Material (D 2139 - 62 T) (Accepted December 17, 1962).

Revision of Tentative Methods for:

Sample Preparation for Physical Testing of Rubber Products (D 15 - 59 T), (one revision accepted April 25, 1962, and the second on September 11, 1962).

Revision of Tentative Methods of Test for:

Change in Properties of Elastomeric Vulcanizates Resulting from Immersion in Liquids (D 471 - 59 T) (Accepted December 17, 1962),

Accelerated Ozone Cracking of Vulcanized Rubber (D 1149 - 60 T) (Accepted September 11, 1962), and

Resistance to Aging of Vulcanized Rubber by Measurement of Creep (D 1206 - 52 T) (Accepted September 11, 1962).

Revision of Tentative Methods for Chemical Analysis of:

Synthetic Elastomers (Solid Styrene-Butadiene Copolymers) (D 1416 - 61 T) (one revision accepted March 8, 1962, and the second on September 11, 1962).

Revision of Tentative Specifications for:

Thermoplastic Vinyl-Chloride Plastic Sheath Compound for Electrical Insulated Cords and Cables (D 1047 - 49 T) (Accepted February 6, 1963),

Nonmetallic Gasket Materials for General and Aeronautical Purposes (D 1170 - 59 T) (Accepted September 11, 1962),

Polyethylene Insulated Wire and Cable (D 1351 - 54 T) (Accepted September 11, 1962),

Synthetic Rubber Heat- and Moisture-Resistant Insulation for Wire and Cable, 75 C Operation (D 1679 - 59 T) (Accepted December 17, 1962), and

Carbon Blacks Used in Rubber Products (D 1765 - 61 T) (Accepted December 17, 1962).

Revision of Tentative Specifications and Methods of Test for:

Sponge and Expanded Cellular Rubber Products (D 1056 - 59 T) (Accepted December 17), 1962, and

Flexible Urethane Foam (D 1564 - 59 T) (Accepted February 6, 1963).

Revision of Tentative Recommended Practice for:

Description of Types of Styrene-Butadiene Rubbers (SBR) and Butadiene Rubbers (BR) (D 1419 - 61 T) (Accepted December 17, 1962), and

Description of Types of Styrene-Butadiene Rubber (SBR) and Butadiene Rubber (BR) Latices (D 1420 - 60 T) (Accepted December 17, 1962).

Revision of Tentative Definitions of:

Terms Relating to Rubber and Rubber-Like Materials (D 1566 - 60 T) (Accepted September 20, 1962).

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee D-11 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] All the existing standards and tentatives referred to in this report appear in their present form in the 1961 Book of ASTM Standards, Part 11.

Adoption of Tentatives as Standard Without Revision

Tentative Method of:

Calibrating a Light Source Used for Accelerating the Deterioration of Rubber (**D 749 - 43 T**).

Tentative Specifications for:

Friction Tape for General Use for Electrical Purposes (**D 69 - 59 T**).

Revision of Standards, Immediate Adoption

(Requires nine-tenth affirmative vote)

Standard Methods of Testing Automotive Air Brake and Vacuum Brake Hose (D 622 - 60):

New Section 12.—After Section 11 under "Air Brake Hose" add a new section entitled "Web Test" to read as follows, renumbering the present Section 12 and subsequent sections accordingly:

12. (*a*) *Web Separation.*—A 10-in. length of hose shall be carefully separated at the point of cleavage between cover stock (web) for a distance of 3 in. The hose shall be pulled apart with a slow steady pull by hand for the remaining 7 in. The lines must separate without exposing the braid.

(*b*) *Web Strength.*—With a joined sample 1 in. long, a rod shall be inserted through the bore of each of the two hoses and the rod suitably fixed in a tension testing machine. The load required to separate the two hoses shall be recorded.

Standard Methods of Test for Contact and Migration Stain of Vulcanized Rubber in Contact with Organic finishes (D 925 - 55):

Section 6(b).—Change the last sentence to read "A new lamp must be broken in for 24 hr before use and shall be used for not over 176 hr thereafter."

Standard Method of Test for Discoloration of Vulcanized Rubber: Organic Finish Coated or Light Colored (D 1148 - 55):

Section 2(a).—Change the last sentence to read: "A new lamp must be broken in for 24 hr before use and shall be used for not over 176 hr thereafter."

Standard Method of Test for Viscosity and Curing Characteristics of Rubber by the Shearing Disk Viscometer (D 1646 - 62):

Section 3(f).—In sentence 4 beginning "Dies closed by hydraulic means. . . ." substitute "pneumatic" for "hydraulic" and substitute "2535 ± 110 lb (1150 ± 50 kg)" for "750 lb." Change sentence 5 to read: "A greater force may be required to close the platens when tests are made on stocks having extreme toughness." In sentence 6 substitute "2535 ± 110 lb (1150 ± 50 kg)" for "750 lb."

Tentatives Continued Without Revision

The committee recommends that the tentatives under its jurisdiction other than those referred to in this report, be continued as tentative. Many are under consideration for revision.

Editorial Change

Standard Methods of Test for Compression Fatigue of Vulcanized Rubber (D 623 - 62):

Section 17(a).—Change the last two sentences to read: "The temperature of the plates shall be held at equilibrium temperature at the start of each test. A temperature of 50 ± 1 C (122 ± 1.8 F) is recommended."

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[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

This report has been submitted to letter ballot of the committee, which consists of 265 members; 185 members returned their ballot, of whom 159 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

G. C. MAASSEN,
Chairman.

J. F. KERSCHER,
Secretary.

REPORT OF COMMITTEE D-12
ON
SOAPS AND OTHER DETERGENTS*

Committee D-12 on Soaps and Other Detergents held one meeting during the year: on March 11 and 12, 1963, in New York, N. Y. The following papers were presented at the meeting: (1) "Automatic Cloud Point Determination for Nonionic Detergent"[1] by H. Baum, T. Callan, and R. Sobatzki; (2) "A Soil Accumulation Method for Comparing Cleaning Power of Household Detergents in the Laboratory"[2] by A. M. Schwartz; and (3) "Analytical Techniques Employed in the Study of Detergent Biodegradation"[3] by R. C. Allred, E. A. Setzkorn, and R. L. Huddleston.

There was considerable discussion on the problem of biodegradable detergents. It was decided that Committee D-12 could serve best by cooperating with Committee D-19 on Industrial Water and with The Soap and Detergent Assn. at this time.

A contribution of $1000 was made by the committee to the ASTM Headquarters building fund.

The committee consists of 161 members, of whom 87 are classified as producers, 42 as consumers, and 32 as general interest members, with 5 consulting members.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee D-12 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[4] All of the existing tentatives and standards referred to appear in their present form in the 1961 Book of ASTM Standards, Part 10, or in the 1962 Supplement thereto.

Proposed Specification and Methods to be Published as Information

as appended hereto[5]

Proposed Specification for Drycleaning Detergent, Non-charge Type,
Proposed Simplified Ion Exchange Method for Analysis of Sodium Triphosphate, and
Proposed Method of Test for Sodium Carboxymethyl Cellulose (CMC) in Detergent Mixtures.

New Tentatives

as appended hereto[6]

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] Being submitted for publication in *Materials Research & Standards.*

[2] Being submitted for publication in *Soap and Chemical Specialities.*

[3] Being submitted for publication in *Journal,* Am. Oil Chemists Soc.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[5] The proposed specification and methods appear in the *1964 Book of ASTM Standards,* Part 22.

[6] The new tentatives appear in the *1964 Book of ASTM Standards,* Part 22.

Tentative Specifications for:

Combination Bar Soap.

Tentative Method of Test for:

Active Oxygen in Bleaching Compounds.

Adoption of Tentatives as Standard Without Revision

Tentative Methods of Sampling and Chemical Analysis of:

Alkylbenzene Sulfonates (**D 1568 - 60 T**), and Fatty Alkyl Sulfates (**D 1570 - 60 T**).

Editorial Changes

Standard Method of Test for Ethylene Diamine Tetraacetate (EDTA) in Soap or Synthetic Detergents (**D 1767 - 62**):

Section 1.—Add "Throughout this method EDTA represents the hydrated disodium salt containing 2 moles of H_2O. In the chemical literature this is frequently abbreviated to $Na_2H_2Y_4 \cdot 2H_2O$."

Section 3(a).—Delete Note 1.

Footnote 5.—Change "No. 6354" to read "No. 7192."

Section 4.—Renumber Note 2 as Note 1.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee T-2 on Analysis of Soaps and Synthetic Detergents (H C. Speel, chairman) through its Task Group 4 (R. M. Kelley, chairman) has been working on methods for the separation of active ingredients. After discussion with Task Groups 5 (E. A. Setzkorn, chairman) and 16 (John Duffy, chairman) it was decided to split the proposed specification into two parts, one on qualitative separations and the other on quantitative ones. Task Group 5 is rewriting a method for the infrared determination of surfactants. Task Group 8 (J. D. Knight, chairman) asked to be discharged but submitted a summary of the results that had been obtained on the determination of sodium carboxymethyl cellulose to be published as information, as appended to this report. Task Group 22 (E. W. Blank, chairman) found that a method for the evaluation of ethylene diamine sodium tetraacetate proposed by the British Standards Institution gave inconclusive and erratic results. Task Group 24 (C. H. Schmiege, chairman) submitted the new Tentative Method for Determination of Active Oxygen in Bleaching Compounds appended to this report.

Subcommittee T-4 on Analysis of Inorganic Alkaline Detergents (M. V. Trexler, chairman) investigated the reproducibility of modified methods of analysis of sodium triphosphate. Task Group 1 (R. J. Fuchs, chairman) found that the modified Paper Chromatographic Method published as information last year was faster than that in the present Tentative Methods D 501. However, certain details need clarification before the proposed method can be issued as tentative. Task Group 2 (E. Moore, chairman) investigated a direct flow ion-exchange method which takes only 3 hr as compared with 8 hr required for the present reverse flow method in Tentative Methods D 501 - 62 T. It was found necessary to revise the method to improve reproducibility. The revised method, appended to this report, is being published as information at this time and will be tested to see whether it can be issued as tentative.

Subcommittee S-2 on Specifications for Soaps and Synthetic Detergents (W. H. Joy, chairman) presented the new Tentative Specifications for Combination Bar Soap prepared by Task Group 3 (R. M. Kelley, chairman). Task Group 1 (R. E. Hauber, chairman) has not been able to resolve differing opinions on method of preparation and total active ingredients in a high sudsing detergent but is continuing to work on these problems. Task Group 4 (B. Cohen, chairman) is working on specifications for a general-purpose synthetic detergent of the liquid type.

Subcommittees S-3 on Specifications for Drycleaning, and *T-3 on Analysis of Drycleaning Materials* (A. R. Martin, chairman) prepared the appended Proposed Specification for Drycleaning Detergent, Non-charge Type, published as information and will now try to obtain methods to implement this and the charge-type detergent specification published as information last year.

Subcommittee T-5 on Physical Testing (L. G. Weeks, chairman) has combined Task Group 1, 3, and 6 into a new Task Group 1 on Reflectance, Detergency, and Redeposition (Marvin Mausner, chairman). This group will study fabric detergency. Task Group 5 (J. L. Staubly, chairman) has conducted a literature survey and submitted material to bring the Bibliographic Abstracts on Evaluation of Brightening Agents for Detergent Usage (*STP 177*) up to date. A cooperative test will be conducted to evaluate several brighteners incorporated into a detergent base for substantivity to cloth and build-up during washing. Task Group 8 on rug cleaning is being reactivated. Task Group 10 (R. E. Johnson, chairman, J. Dupré, acting chairman) ran cooperative tests on foam measurement in a dishwashing machine and got good agreement between laboratories. Further tests will be made to compare detergents more closely in foaming properties, with different soils and different washing machines.

This report has been submitted to letter ballot of the committee, which consists of 161 members; 88 members returned their ballots, of whom 73 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

WILLIAM STERICKER,
Chairman.

H. R. SUTER,
Secretary.

REPORT OF COMMITTEE D-13

ON

TEXTILE MATERIALS*

Committee D-13 on Textile Materials held two four-day meetings in October, 1962, and March, 1963, in New York, N. Y. At each meeting some 70 subcommittees, task groups, and special committees met to discuss revisions of existing methods and the development of new methods.

At the October meeting the following papers were presented: (1) "Behavior of Filamentous Materials Subject to High Speed Tensile Impact," by Jack C. Smith, National Bureau of Standards; (2) "High Speed Tensile Testing of Single Fibers," by R. W. Singleton, and B. S. Sprague, Celanese Corporation of America; and (3) "Impact Testing of Textile Materials at Velocities up to 700 Feet per Second," by Chauncey Chu and Robert J. Coskren, Fabric Research Laboratories, Inc.

Subcommittee C-6 on Editorial Review and Policy not only continued its efforts to review critically the numerous committee methods to effect uniformity of excellence but it also considered with the staff of ASTM Headquarters changes in the form and recommended practices for writing standards. These efforts are being continued and improved instructions seem very probable, including the use of the modified decimal system for numbering the primary and subsidiary divisions and subdivisions down to individual paragraphs. Committee D-13 feels very strongly that these changes are most desirable and will enhance its methods and standards.

Committee D-13 continued its interests and efforts in the work of International textile standards, especially Pan American standards. Close working relationships have been established with key organizations and individuals in Mexico, Central and South American nations. The United States delegation to the first meetings in Lima, Peru, in November 1962, of the Pan American Standards Committee (PASC) Technical Committee 6 for Textiles reported that an excellent beginning was made as well as plans for future meetings. Juan Villarroel of Peru Agricultural College, Lima, Peru, has become an active member of Committee D-13 and participated in the work of Subcommittee A-3 at its last meeting. This international participation and cooperation is encouraged and should be promoted. Committee D-13 is proud that Werner von Bergen and Burt Johnson, two well-known members, are currently abroad on extended assignments to assist in the use of textile standards and methods. D. S. Hamby and Ruby K. Worner have been on previous assignments and are considering new ones.

Committee D-13 is proud of the fact

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

that the Society gave special recognition to the members of the Task Group on Yarn Appearance Standards for twenty-five years of outstanding achievements. These Cotton Yarn Appearance Standards are used internationally.

At the March meeting of Committee D-13, the Harold DeWitt Smith Memorial Medal for 1963 was presented at a special luncheon to Richard T. Kropf, President of Belding Heminway Co., and a Past-President of the Society, for his outstanding contributions to textile science. Werner von Bergen was elected an Honorary Member of Committee D-13 in recognition of his many years of outstanding contributions to the work of Committee D-13.

D. S. Hamby, first vice-chairman, and H. A. Ehrman, secretary, attended the ASTM meeting of Officers of Technical Committees in Philadelphia, Pa., November, 1962, and presented to ASTM President, A. C. Webber, a check for $1000 in behalf of Committee D-13 for the ASTM Building Fund.

Committee D-13 regrets that after many years of conscientious and dedicated service, Joseph A. Truitt found it necessary to relinquish the chairmanship of Subcommittee B-2 on Definitions and Nomenclature. Miss Josephine M. Blandford has accepted this important assignment. It appears noteworthy that the work of Subcommittee B-2 is under the guidance of two very capable women, Miss Blandford, as chairman, and Miss Genevieve Smith as secretary. The following other appointments have been made during the year: S. C. Mayne, Jr., chairman of Subcommittee A-1 to replace Burt Johnson; W. T. Waters, former secretary was appointed chairman of Subcommittee B-3 to replace S. C. Mayne, Jr., and Travis Hall was appointed secretary; E. E. Berkley, chairman of Subcommittee B-6 to replace Braham Norwick; W. C. Wheatley, chairman of Subcommittee B-8 I; N. B. Gobeil, secretary of Subcommittee B-9 I to replace E. A. McMaster; B. S. Sprague, D-13 representative to Committee E-1 to replace Lyman Fourt; E. E. Berkley, ASTM representative to ASA Committee L-23 to replace Arthur Scroggie, with Arthur Scroggie as alternate to replace Braham Norwick. N. C. Enrick resigned as chairman of Subcommittee B-5, and W. C. Waters resigned as D-13 representative to Subcommittee 14 of Committee E-1. Subcommittee A-10 I has been changed to Subcommittee A-10 on Felt. Subcommittee A-10 II has been changed to Subcommittee A-12 on Nonwoven Fabrics.

The committee is presenting a number of recommendations relating to standards, which are listed in detail in the Appendix.

All other standards and tentatives not specifically referred to are being studied by the respective subcommittees and it is recommended that they be continued in their present status.

This report has been submitted to letter ballot of the committee, which consists of 400 voting members; 247 members returned their ballots, of whom 220 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

H. F. SCHIEFER,
Chairman.

H. A. EHRMAN,
Secretary.

APPENDIX

PROPOSED RECOMMENDATIONS AFFECTING STANDARDS ON TEXTILE MATERIALS

Committee D-13 requests approval by the Society at the Annual Meeting of the recommendations given in this Appendix, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] All of the existing tentatives and standards referred to in this report appear in their present form in the 1961 Book of ASTM Standards, Part 10, and the 1962 Supplement.

New Tentatives

as appended hereto.[2]

Tentative Methods of Test for:

Seed Coat Fragment and Funiculi in Cotton Fiber Samples.

Tentative Tolerances for:

Man-Made Organic Base Filament Single Yarns (a revision and consolidation of Tolerances D 1235, D 1236, and D 1237).

Revision of Tentatives

Tentative Method of Test for Length of Cotton Fibers by Fibrograph (D 1447 – 54 T):

Revise as appended hereto.[2]

Tentative Method of Test for Linear Density of Textile Fibers by the Vibroscope (D 1577 – 60 T):

Revise as appended hereto.[2]

Tentative Method of Test for Durability of Finish of Zippers to Laundering (D 2051 – 61 T):

Section 2.—Change to read as follows retaining the present footnote reference: "The apparatus and materials shall be as specified in AATCC Method 36-1961, Colorfastness to Washing; Characterization of Textile Colorants."

Section 5.—Change the reference to AATCC Method 61-1961 T to read "Method 36-1961."

Tentative Method of Test for Colorfastness of Zippers to Laundering (D 2057 – 61 T):

Sections 2 and 5.—Change as indicated above for Sections 2 and 5 of Method D 2051.

Tentative Revision of Standard

Standard Definitions of Terms Relating to Textile Materials (**D 123 – 62**):

Add as tentative revisions, the new definitions and revised standard definitions as appended hereto.[3]

Revision of Standards, Immediate Adoption

(requires nine-tenths affirmative vote)

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[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[2] The new and revised tentatives appear in the 1963 Supplement to *Book of ASTM Standards*, Part 10.

[3] The tentative revisions appear in the revised standard; see 1963 Supplement to *Book of ASTM Standards*, Part 10.

Standard Definitions of Terms Relating to Textile Materials (D 123 – 62):

Delete the standard definition for Skein, Numbering, *n*, and Note. This definition is being replaced by the new tentative definition Test Skein.

Standard Specifications for Fineness (Grade) of Wool Tops (D 472 – 61):

Table I.—For Grade 62's change the maximum requirement for fibers from "1" to "1.5" for fibers 40.1μ and over.

Standard Methods of Testing Nonwoven Fabrics (D 1117 – 62):

Section 1.—Change to read as follows: "These methods of test are applicable to nonwoven fabrics. A nonwoven fabric is a structure produced by bonding or the interlocking of fibers or both, accomplished by mechanical, chemical, thermal, or solvent means, and the combinations thereof. The term does not include fabrics which are woven, knitted, tufted, or made by wool felting processes.

Standard Methods of Test for Water Resistance of Textile Fabrics (D 583 – 58):

Revise as appended hereto.[4]

Standard Method of Test for Tear Resistance of Woven Fabrics by Falling Pendulum (Elmendorf) Apparatus (D 1424 – 59):

Revise as appended hereto.[4]

Standard Method of Test for Cross-Sectional Characteristics of Cotton Fibers (D 1444 – 56):

Revise as appended hereto.[4]

Adoption of Tentative as Standard With Revision

Tentative Method of Test for Predicting Differential Dyeing Behavior of Cotton (**D 1464 – 57 T**):

Revise as appended hereto.[4]

Adoption of Tentatives as Standard Without Revision

Tentative Definitions of Terms Relating to:

Zippers (**D 2050 – 61 T**).

Tentative Methods of Test for:

Durability of Finish of Zippers to Laundering (**D 2051 – 61 T**),
Colorfastness of Zippers to Drycleaning (**D 2052 – 61 T**),
Colorfastness of Zippers to Light (**D 2053 – 61 T**),
Colorfastness of Zipper Tapes to Crocking (**D 2054 – 61 T**),
Colorfastness of Zipper Tapes to Perspiration (**D 2055 – 61 T**),
Resistance of Finish of Zippers to Dry Abrasion (**D 2056 – 61 T**),
Colorfastness of Zippers to Laundering (**D 2057 – 61 T**),
Durability of Finish of Zippers to Drycleaning (**D 2058 – 61 T**),
Resistance of Zippers to Salt Fog (**D 2059 – 61 T**),
Measuring Zippers (**D 2060 – 61 T**),
Strength Tests of Zippers (**D 2061 – 61 T**), and
Operability of Zippers (**D 2062 – 61 T**).

Revision of Standard and Reversion to Tentative

Tentative Method of Test for Tensile Strength of Wool Fiber Bundles (**D 1294 – 58**): Revise as appended hereto.[2]

Withdrawal of Tentative

Tentative Method of Test for Snag Resistance of Hosiery (**D 1115 – 54 T**).

This method is being withdrawn since it is no longer used.

Withdrawal of Standards

Standard Method of Test for:

Stretch of Hosiery (**D 1058 – 51**).

Standard Tolerances for:

Filament Acetate Yarns (**D 1235 – 58**),
Filament Nylon Yarns (**D 1236 – 58**), and
Filament Rayon Yarns (**D 1237 – 58**).

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[4] The revised standards appear in the 1963 Supplement to *Book of ASTM Standards*, Part 10.

Since only one section of Method D 1058 is being used, and that to a very limited extent, it was decided to withdraw the method. The method may be rewritten around the one section.

Standard Tolerances D 1235, D 1236, and D 1237 are being replaced by the new Tentative Tolerances for Man-Made Organic Base Filament Single Yarns, referred to earlier in this report.

Editorial Changes

Standard Definitions of Terms Relating to Textile Materials (D 123 – 62):

Make editorial changes as appended hereto.[4]

Standard Methods of Testing Felt (D 461 – 61):

Section 19(e).—Add the following new Note 6 at the end of this section, renumbering the present notes accordingly: "NOTE 6.—As an alternate solvent to carbon tetrachloride, 1,1,1, trichlorethane may be used."

Tentative Recommended Practice for Use of the Tex System to Designate Linear Density of Fibers, Yarn Intermediates, Yarns, and Other Textile Materials (D 861 – 62 T):

Appendix VIII.—In Table III change the last figure in the first column under Cotton Count from "5.758" to "5.491."

Standard Methods of Testing Twine Made from Bast and Leaf Fibers (D 1233 – 58):

Section 3(a).—Change the first sentence to read: "Take at random five packages (balls, cones, tubes) each from a different case or carton, if size of shipment permits."

Extensive editorial changes recommended by Subcommittee C-6 on Editorial Review and Policy (A. G. Scroggie, chairman) have been reviewed and approved by the officers of the responsible subcommittees in the following:

Recommended Practice for:

Designation of Yarn Construction (**D 1244 – 58 T**).

Specifications for:

Fineness (Grade) of Wool Tops (**D 472 – 61**), and
Fineness (Grade) of Mohair Tops (**D 1381 – 61**).

Methods of Test for:

Diameter of Wool and Mohair Fibers by Microprojection (**D 2130 – 61**),
pH of Aqueous Extracts of Wool and Similar Animal Fibers (**D 2165 – 61 T**), and
Breaking Load and Elongation of Textile Fabrics (**D 1682 – 59 T**).

The editorial changes will be incorporated in the above standards when they are next published in the 1963 Compilation of ASTM Standards on Textile Materials.

Tentative Methods of Test for Stiffness of Fabrics (D 1388 – 55 T):

Footnote 3.—Change to read as follows:

"The 43-deg angle contained in the earliest instrument manufactured by Fabric Development Tests is a variation from the above specified angle of 41½ deg. When the equipment used contains 43 deg the report will so indicate."

Section 15.—Add a new Paragraph (*e*) to read: "(*e*) Report the angle of the platform of the equipment used (see Section 3 and Footnote 3)."

REPORT OF COMMITTEE D-14

ON

ADHESIVES*

Committee D-14 on Adhesives held two meetings during the year: on Oct. 2 and 3, 1962, in Los Angeles, Calif., and on March 21 and 22, 1963, in Pittsburgh, Pa.

The October meeting was held jointly with Committee C-19 on Structural Sandwich Constructions in continuing common interests on adhesive-adherend problems. Committee D-14 sponsored a Symposium on Adhesives during the Pacific Area Meeting.

At the Pittsburgh meeting, M. E. Lerner of *Adhesives Age* magazine presented the second Committee D-14 award dually to C. H. Dahlquist and D. H. Kaeble in recognition of their contributions to the adhesive field. W. C. Fernelius addressed the committee at this meeting on "Education in Developing Countries."

The committee consists of 145 members, of whom 54 are classified as producers, 57 as consumers, 33 as general interest members, and 1 consultant.

RECOMMENDATIONS FOR SOCIETY ACTION

Committee D-14 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] All of the existing tentatives and standards referred to appear in their present form in the 1961 Book of ASTM Standards, Part 6.

New Tentatives

as appended hereto[2]

Tentative Methods of Test for:

Strength Properties of Metal-to-Metal Adhesives by Compressive Loading (Disk Shear), and

Flow Properties of Adhesives.

Tentative Revision of Standard

Standard Definitions of Terms Relating to Adhesives (**D 907 – 60**):

In the definition of "Time, Assembly" change Item 1 to read "The time between spreading of the adhesive on the adherend(s) and the completion of the assembly of the parts for bonding."

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] EDITOR'S NOTE.—No action was taken at the Annual Meeting on the Report of Committee D-14 in the absence from the session at which the report was scheduled for presentation of an individual authorized to present the report. Alternate procedures were used in securing Society action on the recommendations, set forth herein, as follows:

The Administrative Committee on Standards accepted on Sept. 20, 1963, the two new tentative methods and the tentative revision of Standard Definitions D 907 – 60.

Through the new interim procedure for standards, the Society accepted on Nov. 19, 1963, the revision for immediate adoption of Standard Methods D 1084 – 60.

[2] The new tentatives appear in the 1963 Supplement to *Book of ASTM Standards*, Part 6.

New Definitions.—Add the following four new definitions:

Acceptance Test, *n.*—An investigation performed on an individual lot of a previously qualified adhesive, by, or under the observation of, the purchaser to establish conformity with a purchase agreement.

Qualification Test, *n.*—An investigation, independent of a procurement action, performed on an adhesive product to determine whether or not the product conforms to all requirements of the applicable application.

NOTE.—The examination is usually conducted by the agency responsible for the specification, the purchaser, or by a facility approved by the purchaser, at the request of the supplier seeking inclusion of his adhesive on a qualified products list.

Glue Line, *n.*—An adhesive layer between adherends, also called adhesive line or bond line.

Surface Preparation, *n.*—Any chemical or physical treatment which is applied to or performed on adherends prior to application of the adhesive.

NOTE.—This may be done to improve or decrease adhesive performance.

Revision of Standard, Immediate Adoption

(requires nine-tenths affirmative vote)

Standard Methods of Test for Consistency of Adhesives (**D 1084 – 60**):

Add a new Method D by the Zahn cups, as appended to this report.[3]

Tentatives Continued Without Revision

Tentative Methods of Test For:

Strength Properties of Adhesives in Shear by Tension Loading (Metal-to-Metal) (**D 1002 – 53 T**),

Peel Resistance of Adhesives (T-Peel Test) (**D 1876 – 61 T**),

Nonvolatile Content of Urea-Formaldehyde Resin Solutions (**D 1490 – 57 T**),

Density of Adhesives in Fluid Form (**D 1875 – 61 T**),

Resistance of Adhesives to Cyclic Laboratory Aging Conditions (**D 1183 – 61 T**),

Susceptibility of Dry Adhesive Films to Attack by Roaches (**D 1382 – 55 T**),

Susceptibility of Dry Adhesive Films to Attack by Laboratory Rats (**D 1383 – 55 T**),

Permanence of Adhesive-Bonded Joints in Plywood Under Mold Conditions (**D 1877 – 61 T**),

Pressure-Sensitive Tack of Adhesives (**D 1878 – 61 T**),

Penetration of Adhesives (**D 1916 – 61 T**),

Bonding Permanency for Water- or Solvent-Soluble Liquid Adhesives for Automatic Machine Sealing Top Flaps of Fiberboard Specimens (**D 1713 – 60 T**), and

Water Absorptiveness of Fiberboard Specimens for Adhesives (**D 1714 – 60 T**).

Tentative Recommended Practices for:

Atmospheric Exposure of Adhesive-Bonded Joints and Structures (**D 1828 – 61 T**), and

Exposure of Adhesive Specimens to High-Energy Radiation (**D 1879 – 61 T**).

Tentative Specification for:

Adhesive for Acoustical Materials (**D 1779 – 60 T**).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Strength Tests (G. A. Ilkka, chairman) held two meetings during the year. The subcommittee prepared the New Tentative Method of Test for Strength Properties of Adhesives by Compressive Loading (Disk Shear) appended to this report.[2]

Other methods of test in preparation are for determining torsional shear strength, cleavage strength, and impact strength. Dynamic testing of adhesives at high rates of loading is being considered for future action.

Subcommittee III on Permanence (R. M. Kell, chairman) revised the Method of Test for Resistance of Adhesive Bonds to Chemical Reagents (D 896) to update test procedures and to include reagents which have become important in recent years. This revision is now being balloted by the committee. In other activities, a task force is setting up accelerated and outdoor weathering tests of polysulfide sealants. It is hoped that results of these tests will assist in preparing new test methods for permanence.

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[3] The new Method D of Methods D 1084 is not included in this report but appears in the revised standard; see 1963 Supplement to *Book of ASTM Standards*, Part 6.

Subcommittee IV on Working Properties (L. C. Jackson, chairman) prepared the new Tentative Method of Test for Flow Properties of Adhesives, and the Zahn cup method to be added to Method D 1084, as appended to this report.[2,3]

The proposed method for consistency of thixotropic adhesives had two negative votes in the subcommittee letter ballot. The method is being revised and will be held until Subcommittee VI on Nomenclature has completed its work on pertinent definitions. Further work on the Stormer stroboscopic method of determination of viscosity was dropped as a letter ballot showed no general need for the method.

Subcommittee V on Special Adhesives (Marco Petronio, chairman) prepared the new Specification for Vinyl Acetate Resin Emulsion Adhesive appended to this report. Methods of testing shoe adhesives and optical adhesives have been prepared and are being sent to Subcommittee V for letter ballot.

Subcommittee VI on Nomenclature and Definitions (D. P. Knopf, chairman) submitted five tentative revisions for Definitions D 907 as noted earlier in this report.

Definitions of the following terms have been submitted for committee approval: surface preparation; glue line; viscosity; filler sheet; working or pot life; caul; temperature, maturing; adhesive, solvent.

Subcommittee VI is now working on the definitions for: preproduction inspection; spread; tack bond; adhesive, polar; adhesive, nonpolar; reactivity; and solvent.

Subcommittee VIII on Electrical Properties (H. K. Lauer, chairman).—Most of the work of interlaboratory evalution of the thin film resistance of conductive adhesives using the proposed Committee D-14 test method has been completed. Correlation of results is now under way. The findings have already resulted in revisions concerning surface treatment, thickness determination, jig construction, and method of making electrical contact to the specimen.

The method for determining the bulk electrical properties of a conductive adhesive is still under consideration.

Subcommittee X on Wood Adhesives (R. F. Blomquist, chairman) held a meeting in Pittsburgh, Pa., on March 21, 1963. A principal activity during the year was the development of the first draft of a specification for adhesives for laminated wood products under extreme service conditions, under the chairmanship of W.E. St. Clair. This specification, which was designed to operate on a performance basis rather than by indicating specific chemical composition of the adhesives to insure permanence, was submitted to combined letter ballot of Subcommittee X of Committee D-14 and Subcommittee II of Committee D-7 on Wood. A number of negative ballots will need to be resolved before the revised version is resubmitted to the same combined subcommittee ballot.

A similar specification for adhesives for laminated wood products for interior service is also in the early stages of preparation prior to subcommittee letter ballot.

A revision of Method D 906 has also been under final review to take into account the problem of orientation of lathe checks in the core of the plywood shear test procedure, and a review of different methods to determine free formaldehyde in particle board binders.

Subcommittee XII on Adhesives for Plastics (G. C. Grimes, chairman).—At the 1962 fall meeting, Subcommittee XII was organized into five sections with chairmen picked for four of them. H. Lauer of Hughes Aircraft Co., reported on attempts at plastic-to-plastic peel testing.

The 1962 spring meeting resulted in approval to write a proposed double lap shear test method for plastics.

Harlan Goplen of the Martin Co. is to report on methods of tension testing at the 1963 fall meeting in Boston.

M. J. Bodnar, chairman of Section B on Surface Preparations is studying a report on surface preparations for Mylar (polyethylene terephthalate).

An up-to-date survey of the needs of plastics in bonding is to be made by the subcommittee chairman and presented at the Boston meeting.

Subcommittee XIII on Flooring Adhesives (N. R. Migdol, chairman) conducted an interlaboratory study of a proposed method to determine the strength in tensile shear of floor tile adhesives, above grade. The variability of the results of this study indicated that the test is not very meaningful in its present form. Therefore, a peel strength test will also now be considered along with the tensile shear test which will be revised to account for its limitations. A new interlaboratory study is being initiated to study the new revisions.

This report has been submitted to letter ballot of the committee, which consists of 145 members; 105 members returned their ballots, of whom 93 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

N. J. DELOLLIS,
Chairman.

G. T. BUTTS,
Secretary

REPORT OF COMMITTEE D-15
ON
ENGINE ANTIFREEZES*

Committee D-15 on Engine Antifreezes did not meet during this report period. Subcommittee meetings were held on Oct. 31 and Nov. 1, 1962, in Chicago, Ill. The annual meeting will be held April 23 and 24, 1963, in Washington, D. C.

The committee now has 57 participating individuals, representing 44 voting members, of whom 18 are classified as producer, 15 as consumer, and 11 as general interest members.

Committee D-15 has announced the policy that, where feasible, all new standards under its jurisdiction will indicate values in both the metric and English systems.

RECOMMENDATIONS FOR SOCIETY ACTION

Committee D-15 requests approval by the Society at the Annual Meeting of the following recommendations. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] All of the existing methods referred to in this report appear in their present form in the 1961 Book of ASTM Standards, Part 8.

Revision of Tentative

Tentative Method of Test for Effect of Antifreeze Solutions on Organic Finishes for Automotive Vehicles (**D 1882 - 61 T**):

Section 6.—Add a new note at the end of the section to read: "Stains may disappear after a short exposure to the weather; for example, after 8 to 24 hr of sunshine. Such stains are considered by some persons to be unobjectionable.

In the last line of Paragraph (*b*), add the metric unit "(2.5 cm)" following "1 in."

Section 4.—In the first sentence, insert "(10 by 15 cm)" following "4 by 6 in." In the second sentence, add "(0.25 to 0.50 microns)" after "10 to 20 microinches."

Revision of Standard and Reversion to Tentative

Standard Method of Test for Reserve Alkalinity of Concentrated Engine Antifreezes (**D 1121 - 54**):

Revise and revert to tentative as appended hereto.[2] The method has been expanded to include diluted engine antifreeze.

Editorial Changes

Standard Method of Test for Boiling Point of Engine Antifreezes (**D 1120 - 53**):

Add metric units parenthetically in Sections 3(*d*), 7(*c*), 8(*a*) and (*b*), and in Table I and its footnotes.

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] EDITOR'S NOTE.—No action was taken at the Annual Meeting on the Report of Committee D-15 in the absence of a quorum at the session when the report was scheduled for presentation. Society action on the recommendations, set forth herein, was subsequently taken through the Administrative Committee on Standards. Approval was given to the recommendations by the Standards Committee on Sept. 20, 1963.

[2] The revised tentative appears in the *1964 Book of ASTM Standards*, Part 22.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Hydrometers and Thermometers (R. W. Kallgren, chairman) recommended that Method D 1124 be submitted for approval as American Standard, after editorial addition of metric units in the method.

Subcommittee III on Physical Properties (C. M. White, chairman) approved the reprinting of Method D 1120 with metric units added to the appropriate sections. They are also recommending the addition of a Note to Method D 1882, as detailed above, and are considering a change in the title to include inhibited water coolants.

Subcommittee IV on Chemical Properties (E. F. Harford, chairman) has been continuing work on the expansion of Method D 1121 to include diluted used and unused engine antifreezes, and the revised method is appended to this report.

Subcommittee VI on Simulated and Actual Service Testing (C. H. Sweatt, chairman) is considering the possibility of modifying the metal specimen cleaning procedures of Method D 1384 so that, if certain modifications are also made in Methods B 185 and B 192, all three would agree. This study was requested by the Advisory Committee on Corrosion. The coverage of corrosion inhibitors for water is also under study.

Study Group on Glassware Test Methods (J. G. Willard, chairman) is investigating the possibility of improving solder test coupons by using solder-coated brass. Interlaboratory tests are in progress. The group continues to police the quality of standard specimens.

Study Group on Simulated Service Test Methods (V. O. Hatch, acting chairman) is circulating a simulated service test method for the approval of Subcommittee VI.

Study Group on Engine Dynamometer Test Methods (H. J. Hannigan, chairman) has sent out questionnaires designed to furnish direction in the development of a test method.

Study Group on Vehicle Road Test Methods (M. S. Boehmer, chairman) was organized at the fall meeting, and has a questionnaire circulating which is designed to furnish direction in the development of test procedures. This group will hold its first meeting in April, 1963.

Study Group on Revision of STP No. 120, Selection and Use of Engine Antifreezes (C. M. White, chairman) has revised this work and has circulated a final revision for comment before submission to Committee D-15 for approval.

This report has been submitted to letter ballot of the committee, which consists of 44 voting members; 37 members returned their ballots, all of whom have voted affimatively.

Respectfully submitted on behalf of the committee,

R. E. VOGEL,
Chairman.

W. D. MCMASTER,
Secretary.

REPORT OF COMMITTEE D-16
ON
INDUSTRIAL AROMATIC HYDROCARBONS AND RELATED MATERIALS*

Committee D-16 on Industrial Aromatic Hydrocarbons and Related Materials and its subcommittees held two meetings during the year: on June 27 to 29, 1962, in New York, N. Y., and on Jan. 30 to Feb. 1, 1963, in Tulsa, Okla.

The committee consists of 97 members, of whom 75 are voting members; of these, 37 are classified as producers, 30 as consumers, and 8 as general interest members.

Committee D-16 recommended to the Subcommittee on Thermometers of Committee E-20 on Temperature Measurement the adoption of a new ASTM partial immersion distillation thermometer with a range of 170 to 250 C.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1962 Annual Meeting, Committee D-16 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Methods of Test for:

Aldehydes in Styrene Monomer (D 2119 – 62 T),
Inhibitor, 4-Tertiary-Butylcatechol, in Styrene Monomer (D 2120 – 62 T),
Polymer Content of Styrene Monomer (D 2121 – 62 T),
Assay of Phenol-Water Mixture (D 2145 – 63 T), and
Detection and Estimation of Water Insoluble Impurities in Refined Phenol by Cloud Point Depression (D 2147 – 63 T).

Tentative Methods D 2119, D 2120, and D 2121 were accepted by the Standards Committee on Oct. 22, 1962, and appear in the 1962 Supplement to Book of ASTM Standards, Part 8. Tentative Methods D 2145 and D 2147 were accepted by the Standards Committee on March 21, 1963, and are available as separate reprints.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee D-16 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] All of the existing standards referred to appear in their present form in the 1961 Book of ASTM Standards, Part 8.

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Proposed Method to be Published as Information

Proposed Method for Xylene Isomer Analysis by Gas Chromatography.[2]

Revision of Tentative

Tentative Method for Calculation of Volume and Weight of Benzene, Toluene, and Paraxylene (**D 1555 – 58 T**):

Revise as appended hereto.[3,4]

Adoption of Tentative as Standard Without Revision

Tentative Method of Test for Water in Phenol and Related Materials by the Iodine Reagent Method (**D 1631 – 59 T**).

Withdrawal of Standards and Tentative

The following specifications are recommended for consolidation with identical specifications prepared originally by Committee D-1 for use by the paint industry but which have recently been broadened for general use, namely, Specifications D 361, D 362, and D 364. The specifications will be under the joint jurisdiction of Committees D-1 and D-16.

Specifications for:

Industrial 90 Benzene (**D 837 – 56 T**),
Industrial Grade Toluene (**D 842 – 50**), and
Industrial Grade Xylene (**D 844 – 50**).

Tentatives Continued Without Revision

Tentative Methods of Test for:

Traces of Thiophene in Benzene Using Isatin and Spectrophotometry (**D 1685 – 61 T**), and
Solidification Point of Aromatic Hydrocarbons and Related Materials (**D 1493 – 58 T**).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee A on Monocyclic Aromatics (K. M. Brown, chairman) has appointed a new task group on revision of specifications and specifications for new grades of products within its scope. Another new task group will cooperatively test and compare the hydrometer and Lipkin pycnometer methods for specific gravity. The styrene task group is studying methods for peroxide, sulfur, and chloride contents and purity of styrene. Other task groups are making progress in determination of trace sulfur, non-aromatics by gas-liquid chromatography, and traces of carbon disulfide in benzene by spectrophotometry. This subcommittee is responsible for the revision of Method D 1555 and the new Proposed Method of Test for Xylene Isomer Analysis by Gas Chromatography, as appended to this report.[2,3]

Subcommittee B on Polycyclic Aromatics (R. F. Shertzer, chairman) has developed and cooperatively tested a method for evaporation residue of naphthalene. A visual method for acid wash color of refined naphthalene is ready for cooperative testing.

Subcommittee C on Phenolic Compounds (D. F. Pontz, chairman) developed the new methods for cloud point depression and bromide-bromate assay of phenol. The task group on tar acids is working on methods for pyridine bases and gel-time. A task group on phenol handling and sampling is active under Subcommittee J. Data on gas chromatographic analysis of phenols are being collected. Stability of platinum-cobalt color standards is undergoing study.

Subcommittee D on Nitrogen Hetero-

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[2] The proposed method appears in the *1964 Book of ASTM Standards*, Part 20.

[3] The revised tentative appears in the *1964 Book of ASTM Standards*, Part 20.

[4] A discussion of the development of the volume correction factors in this method is appended; see p. 402.

cyclics (C. K. M. Winne, chairman) has developed methods for water solubility of and reducing substances in refined pyridine.

Subcommittee E on Thermometry (M. Lapeyrouse, chairman) has made recommendations on a tar acid distillation thermometer and is considering a revision of Method D 1493.

Subcommittee F on Editorial and Nomenclature (E. T. Scafe, chairman) has served as a consultant body to aid the other subcommittees when editorial problems are involved.

Subcommittee G on Statistical Procedure (W. J. Tancig, chairman) has served as a consultant body to aid the other subcommittees when statistical problems are involved.

Subcommittee H, American Group ISO/TC 78 on Aromatic Hydrocarbons (K. M. Brown, chairman) sent two delegates to the Fourth Plenary Meeting of ISO/TC 78 at the Hague, Netherlands, in June, 1962.

Subcommittee I on Phthalic Anhydride (O. E. Knapp, chairman) has completed cooperative testing of methods for color molten, heat color, and solidification point of phthalic anhydride and has enlarged its scope to include maleic anhydride.

Subcommittee J on Handling and Sampling (W. J. Tancig, chairman) has reviewed the ISO/TC 78 sampling procedure for aromatic hydrocarbons and is working with Subcommittee C to devise a recommended practice for handling phenol.

This report has been submitted to letter ballot of the committee, which consists of 75 voting members; 57 members returned their ballots, of whom 50 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. E. SISCO,
Chairman.

K. H. FERBER,
Secretary.

APPENDIX

DEVELOPMENT OF VOLUME CORRECTION FACTORS APPEARING IN ASTM METHOD D 1555[1]

The basis for calculating the volume correction factors appearing in the Method for Calculation of Volume and Weight of Benzene, Toluene, and Paraxylene (ASTM Designation D 1555)[1] is described.

Basic Data

For all materials listed, except styrene, density data for pure hydrocarbons were used. For styrene, data for 99.7 per cent purity material were used. All such data are listed in Table I.

By means of a least squares procedure using orthogonalized polynomials,[2] equations of the following type were obtained; all computations were carried out in excess of eight digits:

$$d_t = \delta_o + \alpha t + \beta t^2 + \gamma t^3 \ldots\ldots (1)$$

where:

d_t = density, grams (mass) per milliliters at temperature t,

δ_o = constant term (also density at 0 C for substances liquid at that temperature),

α, β, γ = linear, quadratic, and cubic coefficients, and

t = temperature, deg C.

In the least squares solution, up to the fourth order power series equation was developed, the root means square error terms for each coefficient being obtained as well. When the highest order coefficient in a given order equation failed to pass the t-test, that order equation was discarded. It is for this reason that the number of coefficients in Table I is variable.

It was assumed that all density values were reliable to a few units in the fifth decimal place; to minimize influence of rounding in the final data, seven decimal places were assigned to the constant term and correspondingly more to the higher order coefficients of temperature.

The δ_o term was then adjusted so as to give a value of d_{20} exactly equal to those given in Table II so as to match the values given in the tables sponsored by API Project 44.[3]

Final values for all basic equation constants used subsequently are listed in Table II.

Volume Reduction to 60 F

Equation 1 in the Celsius scale was converted to the following form in the Fahrenheit scale with correspondingly altered constants:

$$D_T = D_o + AT + BT^2 + CT^3 \ldots (2)$$

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[1] Tentative Method for Calculation of Volume and Weight of Benzene, Toluene, and Paraxylene (D 1555 - 63 T); *1964 Book of ASTM Standards*, Part 20.

[2] P. G. Guest, "Orthogonal Polynomials in the Least Squares Fitting of Observations," *Philosophical Magazine*, Vol. 41, p. 124 (1950).

[3] American Petroleum Institute, Research Project 44. Maintained at Chemical Thermodynamic Properties Center, Dept. of Chemistry, Agricultural and Mechanical College of Texas, College Station, Texas.

TABLE I.—AVAILABLE DENSITIES.

Temperature, deg C (t C)	Density, g per ml (d_t)					t C	d_t
	Benzene[a]	Toluene[a]	*o*-Xylene[a]	*p*-Xylene[a]	*m*-Xylene[a]	Styrene[b]	
−10	...	0.89461	0.90521	...	0.88949	0	0.92390
0	...	0.88538	0.89690	...	0.88107		0.92384
10	0.88947	0.87615	0.88856	0.86966[c]	0.87263	7	0.91771
20	0.87901	0.86692	0.88020	0.86103	0.86414		0.91771
30	0.86845	0.85769	0.87180	0.85239	0.85563	14	0.91160
40	0.85774	0.84846	0.86336	0.84372	0.84705		0.91159
50	0.84688	0.83923	0.85489	0.83499	0.83842		0.91158
60	0.83588	0.83000	0.84637	0.82618	0.82972	21	0.90538
70	0.82473	0.82077	0.83780	0.81731	0.82096		0.90538
80	0.81343	0.81154	0.82915	0.80834	0.81216		0.90536
90	...	0.80231	0.82041	0.79930	0.80330	28	0.89912
100	...	0.79308	0.81155	0.79019	0.79329		0.89910
							0.89909
						35	0.89290
							0.89290
							0.89289
						42	0.86672
							0.88670
							0.88669
						49	0.88047
							0.88046
							0.88045

[a] Data for 100 per cent purity material; supplied by the director of API Project 44 (December 2, 1955).

[b] Data for 99.7 per cent molal purity product, supplied by Dow Chemical Co., Midland, Mich. (June 17, 1960).

[c] For undercooled liquid.

TABLE II.—BASIC COEFFICIENTS.

Material	d_{20}	δ_0	α	β	γ
Benzene	0.87901[a]	0.8997261	−0.001021458	−0.00000071726	...
Toluene	0.86696[a]	0.8854200	−0.000923000	...	...
o-Xylene	0.88020[a]	0.8969025	−0.000833507	−0.00000005180	−0.0000000014556
m-Xylene	0.86417[a]	0.8809567	−0.000831026	−0.00000041548	...
p-Xylene	0.86105[a]	0.8782324	−0.000856634	−0.00000009580	−0.0000000014258
Mixed Xylenes	...	0.8809567	−0.000831026	−0.00000041548	...
Styrene	0.90624[b]	0.9238927	−0.000880293	−0.00000012904	...

[a] Taken from Table 5a of "Selected Values of Properties of Hydrocarbons and Related Compounds" (Feb., 1962) prepared by American Petroleum Institute, Research Project 44 (see Footnote 3).

[b] Rounded value from equation constants.

where:

D_T = density, grams per milliliter at temperature T,

$D_o = d_o - (\frac{5}{9})(32)\alpha + (\frac{5}{9})^2(32)^2\beta - (\frac{5}{9})^3(32)^3\gamma$,

$A = + (\frac{5}{9})\ \alpha + 2(\frac{5}{9})^2(32)\ \beta + 3(\frac{5}{9})^3(32)^2\gamma$,

$B = + (\frac{5}{9})^2\beta - 3(\frac{5}{9})^3(32)\ \gamma$,

$C = + (\frac{5}{9})^3\gamma$, and

T = temperature, deg F.

Volume correction multipliers were calculated at 1 F intervals by means of the following relationship:

$$Multiplier = V_{60}/V_T = D_T/D_{60} \quad \text{....(3)}$$

Pounds per gallon for *p*-xylene were

calculated from:

$$\text{Apparent density } P = \left[\frac{V_s}{W_s}\right] \frac{(D_T - d_a)}{1 - (d_a/d_b)} \quad (4)$$

where:

P = pounds in air per U.S. gallon at temperature T, deg F,

D_T = density of hydrocarbon, grams per milliliter at temperature T, deg F (Eq. 2),

d_a = assumed mean density of air, 0.0012 g per ml,[4]

d_b = assumed mean density of weights, 8.332 g per ml,[4]

V_s = 3785.306 ml per U.S. gal,[5] and

W_s = 453.59243 g per lb.[5]

All computations were carried out with an excess of eight non-zero digits, rounding to list (by adding five to the last printed digit plus one and then truncating the sum at that point) being performed only as the last operation before typeout.

ASSESSMENT OF WORKING DATA

Benzene and Toluene.—Volume correction tables of the type listed in Method D 1555 were prepared for a number of typical compositions in the range of 95 to 100 per cent purity. Almost no values exceeded those for the pure materials by 0.0001 except at the highest and lowest temperatures, and most were less than that amount. Hence, values for pure compounds were used.

p-Xylene.—Complete volume correction tables and pounds per gallon tables were prepared for several typical compositions in the range of 94 to 100 per cent purity. Almost no volume correction multiplier exceeded 0.0001 or pounds per gallon value exceeded 0.001 except at extreme temperatures listed (above 100 F) as compared to that for pure *p*-xylene. Hence data for the latter were used.

o-Xylene, m-Xylene.—Based on the experience with *p*-xylene, the data for the pure compound were expected to apply to commercial product.

Mixed Xylenes.—A very large number of typical commercial mixtures were compared with pure *m*-xylene and, while pounds per gallon values varied widely, volume correction factors were in close agreement (0.0001) with those for pure *m*-xylene. At temperatures above 100 F and below 30 F, larger differences were observed with none exceeding 0.0004.

Styrene.—Data for a typical product having a molal purity of 99.7 per cent were used and considered applicable for a wider range of purities.

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[4] Derived from "Supplement for Precise Laboratory Standards of Mass," National Bureau of Standards, *Form 109*, revised June 3, 1958.

[5] Obtained from "Units of Weight and Measure," National Bureau of Standards, *Miscellaneous Publication 233*, Dec. 20, 1960.

REPORT OF COMMITTEE D-17
ON
NAVAL STORES*

Committee D-17 on Naval Stores held two meetings during the past two years: on May 3, 1962, in Jacksonville, Fla. and on April 23, 1963, in Atlanta, Ga. The meetings were held jointly with the Technical Committee, Tall Oil Division of the Pulp Chemicals Assn.

The committee reorganized the subcommittees in order to reflect the change in industry practices in the production of naval stores as follows: Subcommittee I on Rosin, Subcommittee II on Terpene Hydrocarbons and Pine Oil, Subcommittee III on Tall Oil, Subcommittee IV on Nomenclature and Definitions, and Subcommittee V on Specifications.

The committee consists of 23 members, of whom 11 are classified as producers, 6 as consumers, and 6 as general interest members.

RECOMMENDATIONS FOR SOCIETY ACTION

Committee D-17 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] The existing tentative and standard referred to appear in their present form in the 1961 Book of ASTM Standards, Part 8.

Adoption of Tentative as Standard With Revision

Tentative Methods of Test for Fatty Acids Content of Tall Oil Rosin (**D 1585 – 58 T**):

Section 9(a).—Change reflux time from "5 min" to read "2 min."

Revision of Standard, Immediate Adoption

(requires nine-tenths affirmative vote)

Standard Definitions of Terms Relating to Naval Stores and Related Products (**D 804 – 57**):

New Definitions.—Add new definitions as appended hereto[2] in their proper alphabetical order.

This report has been submitted to letter ballot of the committee, which consists of 23 members; 20 members returned their ballots, of whom 16 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

D. D. Mowat,
Chairman.

J. L. Boyer,
Secretary.

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] Editor's Note.—No action was taken at the Annual Meeting on the Report of Committee D-17 in the absence of a quorum at the session when the report was scheduled for presentation. Society action on the recommendations set forth herein was subsequently taken through the new interim procedure for standards. Approval was given to the recommendations by the Society on Nov. 19, 1963.

[2] The new definitions appear in the revised standard; see *1964 Book of ASTM Standards*, Part 20.

REPORT OF COMMITTEE D-18
ON
SOILS FOR ENGINEERING PURPOSES*

Committee D-18 on Soils for Engineering Purposes held a meeting in New York, N. Y., on June 26, 1962, an Executive Committee meeting in New York, N. Y., on June 24, and a meeting in Los Angeles, Calif., on Oct. 5, 1962, during the Fourth Pacific Area National Meeting.

A two-session Symposium on Field Tests and Measurements for Soils and Foundation Engineering[1] was held at the Annual Meeting in June, 1962.

Three sessions were held during the Fourth Pacific Area National Meeting, Sept. 30 to Oct. 5, 1962: one session on land slides, one on subsidence, and a third on general soil subjects. The papers presented at these sessions are being published with the Symposium on Field Tests.[1]

The committee records with sorrow the deaths of Benjamin A. Anderson in August, 1962, and Donald T. Davidson in October, 1962.

The following appointments were made during the year: D. M. Burmister as representative on ASA Section Committee Z84 on Glossary of Environmental Terms; Carl Crawford as liaison member with British Standards Inst.; and John Gnaedinger as representative on Committee E-10 on Radioisotopes and Radiation Effects.

The committee consists of 166 members (118 voting members, of whom 24 are classified as producer, 36 as consumer, and 58 as general interest members).

A three-session Symposium on Soil Exploration is scheduled for June 23 and 24 during the 1963 Annual Meeting in Atlantic City, N. J. with A. I. Johnson and J. P. Gnaedinger as co-chairmen of the symposium.

R. K. Bernhard was selected to receive the Hogentogler Award at the 1963 Annual Meeting for his paper "Biaxial Stress Fields in Noncohesive Soils Subjected to Vibratory Loads."

A Joint Meeting of Committee D-18 and the National Research Council of Canada will be held in Ottawa, Canada on Sept. 9 to 11, 1963. C. B. Crawford is chairman of arrangements for this meeting. Papers will be presented on Laboratory Shear Testing of Soils.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee D-18 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[2] All of the existing tentatives and standards referred to appear in their present form in the 1961 Book of ASTM Standards, Part 4, or the 1962 Supplement thereto.

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* Sixty-sixth Annual Meeting of the Society, June 23 - 28, 1963.

[1] Published as *ASTM STP 322*.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

New Tentatives

as appended hereto.[3]

Tentative Method for:

Calibration of Mechanical Laboratory Soil Compactors.

Tentative Methods of Test for:

Unconfined Compressive Strength of Cohesive Soils, and

Density of Soil in Place by the Rubber Balloon Method.

Revision of Tentatives

Tentative Method for Soil Investigation and Sampling by Auger Borings (D 1452 - 56 T):

Section 1(b).—Change the second sentence to read: "They may be used for any purpose where disturbed samples can be used and are valuable in connection with ground water level determination and indication of changes in strata and advancement of hole for spoon and tube sampling."

Section 3(c).—Change the third sentence to read: "Seal all samples in a jar or other airtight container and label appropriately."

Section 4.—Change to read as follows:

> 4. The data obtained in boring shall be recorded in the field logs and shall include the following:
>
> (*1*) Date of start and completion of boring,
>
> (*2*) Identifying number of boring,
>
> (*3*) Reference datum including direction and distance of boring relative to reference line of project or other suitable reference points,
>
> (*4*) Depths to changes in strata,
>
> (*5*) Diameter and method of advancing bore hole,
>
> (*6*) Description of soil in each major stratum,
>
> (*7*) Ground water elevation and location of seepage zones when found, and
>
> (*8*) Condition of augered hole upon removal of auger—whether the hole remains open or the sides cave, when such can be observed.

Tentative Method for Penetration Test and Split-Barrel Sampling of Soils (D 1586 - 58 T):

Section 2(a).—After the first sentence, add three new sentences to read as follows:

> The stiffness of the drill rod used during sampling is believed to have a relationship to the N value obtained, especially because a light drill rod "whips" under the blows of the hammer. It is suggested that the drill rod have a stiffness equal to or greater than the A-rod. A stiffer drill rod is suggested for holes deeper than 50 ft.

At the end of Paragraph (*a*), reference a new footnote to read: "M. J. Hvorslev, "Subsurface Exploration and Sampling of Soils for Civil Engineering Purposes," *The Engineering Foundation*, 345 East 47th Street, New York 17, New York."

Section 3.—Change the heading of Paragraph (*a*) to read "*Preparing Hole*," and after the first sentence add: "In saturated sands and silts withdraw the drill bit slowly to prevent loosening of the soil around the hole."

Change Paragraph (*b*) to read as follows:

> (*b*) *Obtaining Samples*.—With the sampler resting on the bottom of the hole, seat the sampler 6 in. with blows from the 140-lb hammer falling 30 in. Proceed with driving the sampler until it has been driven an additional 12 in., or until 100 blows have been applied. Record the number of blows required to effect each 6 in. of penetration. The first 6 in. of drive is considered to be a seating drive. The total number of blows required for the second and third 6 in. of penetration shall be termed the penetration resistance, N. If the sampler is driven less than 18 in. total, the penetration resistance shall be for the last foot of penetration (if less than 1 ft is penetrated, the logs shall state the number of blows and the fraction of a foot penetrated). If specified, more than 100 blows may be used for specific types of work. Refusal shall be considered to have been reached when the rate of advance is less than 1 in. for 50 blows.

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[3] The new tentatives appear in the 1963 Supplement to *Book of ASTM Standards*, Part 4.

In Paragraph (*d*) change "24 hr until" to read "24 hr after" in the last sentence.

Figure 1.—Change the 24-in. minimum dimension to apply from the pointed end of the driving shoe to the vent holes, and designate the thread at the right-hand end of the coupling as "A-Rod Thread."

Change Note 1 below Fig. 1 to read: "A coupling head with a check valve and a minimum of 0.6 sq in. venting to outside above check valve may be substituted for coupling shown above to improve sample recovery."

Tentative Method for Thin-Walled Tube Sampling of Soils (D 1587 – 58 T):

Section 1(b).—In the first sentence change "piston-type samplers" to read "piston samplers."

Section 2(b).—Change the first sentence and add a new second sentence to read as follows:

> Tubes 2 to 5 in. in outside diameter and made of any materials having adequate strength and resistance to corrosion will be satisfactory (Fig. 1). Adequate resistance to corrosion can be provided by a suitable coating.

After the sentence "Two vent holes . . . sampler head," add the following new sentence: "A coupling head with a check valve and a minimum of 0.6 sq in. venting to outside above check valve can be used to improve sample recovery."

Section 3.—Change the heading in Paragraph (*a*) to read "*Preparing Hole*," and after the first sentence add: "In saturated sands and silts withdraw the drill bit slowly to prevent loosening of the soil around the hole."

In the next to last sentence of Paragraph (*d*), delete the words "sawdust or other suitable material as packing" and substitute the words "suitable resilient packing material to reduce shock."

In the next to last sentence in Paragraph (*e*), change "24 hr until" to read "24 hr after."

Table I.—Change to read as shown in the accompanying Table I.[4]

Adoption of Tentatives As Standard Without Revision

Tentative Methods of Test for:

Moisture-Penetration Resistance Relations of Fine-Grained Soils (**D 1558 – 58 T**),

Making and Curing Soil-Cement Compression and Flexure Test Specimens in the Laboratory (**D 1632 – 59 T**),

Compressive Strength of Molded Soil-Cement Cylinders (**D 1633 – 59 T**),

Compressive Strength of Soil-Cement Using Portions of Beams Broken in Flexure (Modified Cube Method) (**D 1634 – 59 T**),

Flexure Strength of Soil-Cement Using Simple Beam with Third-Point Loading (**D 1635 – 59 T**).

Tentatives Continued Without Revision

Tentative Methods of Test for:

Shear Strength of Flexible Road Surfaces, Subgrade, and Fills by the Burggraf Shear Apparatus (**D 916 – 47 T**),

Water Soluble Chlorides Present as Admixtures in Graded Aggregate Road Mixtures (**D 1411 – 56 T**),

Density of Soil in Place by the Sand-Cone Method (**D 1556 – 58 T**),

Diamond Core Drilling for Site Investigation (**D 2113 – 62 T**),

Liquid Limit of Soils (**D 423 – 61 T**),

Moisture-Density Relations of Soils Using 5.5-lb Rammer and 12-in. Drop (**D 698 – 58 T**),

Moisture-Density Relations of Soils Using 10-lb Rammer and 18-in. Drop (**D 1557 – 58 T**),

Bearing Ratio of Laboratory-Compacted Soils (**D 1883 – 61 T**), and

Load-Settlement Relationship for Individual Piles Under Vertical Axial Load (**D 1143 – 61 T**).

Tentative Definitions of Terms Relating to:

Soil Dynamics (**D 1707 – 60 T**).

Tentative Specifications for:

Materials for Soil-Aggregate Subbase, Base, and Surface Courses (**D 1241 – 55 T**).

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[4] Table I is not included in this report but appears in the revised method; see 1963 Supplement to *Book of ASTM Standards*, Part 4.

Editorial Changes

Standard Method for Repetitive Static Load Tests of Soils, for Use in Evaluation and Design of Airport and Highway Pavements (**D 1195 - 57**), and Standard Method for Nonrepetitive Static Load Tests of Soils, for Use in Evaluation and Design of Airport and Highway Pavements (**D 1196 - 57**):

Make the following editorial changes in both methods:

Title and Section 1.—Following the word "Static" insert the word "Plate." Following the word "Soils" insert the words "and Flexible Pavement Components."

Section 2.—In Paragraph (*c*) add the following sentence: "The diameters of adjacent plates in the pyramid arrangement shall not differ by more than 6 in."

In Paragraph (*e*) change the last sentence and add a new sentence to read as follows:

> It shall be at least 18 ft long and shall rest on supports located at least 8 ft from the circumference of the bearing plate or nearest wheel or supporting leg. The entire deflection measuring system shall be adequately shaded from direct rays of the sun.

Section 3.—In Paragraph (*a*) add the following new sentence: "To prevent loss of moisture from the subgrade during the load test, cover the exposed subgrade to a distance of 6 ft from the circumference of the bearing plate with a tarpaulin or waterproof paper."

Change Paragraph (*c*) to read as follows:

> (*c*) Use a sufficient number of dial gages, so located and fixed in position as to indicate the average settlement of the bearing plate. When using two dial gages, they shall be set near each extremity of a diameter of the bearing plate, 1 in. from the circumference. When three gages are employed, they shall be set at an angle of 120 deg from each other, and equidistant from the circumference of the bearing plate. Each individual set of readings shall be averaged, and this value is recorded as the average settlement reading.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee G-1, Editorial (E. E. Bauer, chairman) has reviewed editorially the three new tentatives appended to this report and the revised Methods D 1452, D 1586, and D 1587 noted earlier in this report. The subcommittee has also reviewed the Proposed Recommended Practice for Soil-Bituminous Stabilized Base Course for Roads and Streets, the Proposed Method for Laboratory Determination of Moisture Content of Soil, and the Proposed Method for Wet Preparation of Soil Samples for Grain-Size Analysis and Determination of Soil Constants.

Subcommittee G-3 on Nomenclature for Soil Mechanics and Foundations (R. G. Ahlvin, chairman) continues its functions as part of the Joint ASCE-ASTM Committee on Nomenclature in Soil Mechanics and Foundations. The joint committee is reviewing present Definitions D 653 and extending definition coverage into peripheral areas, such as physicochemical properties, geology, physiography, pedology, etc.

As a subcommittee, the group has reviewed a number of standards and tentatives for conformance with the definitions in Standard Definitions of Terms and Symbols Relating to Soil Mechanics (D 653).

The subcommittee has also voted to adopt the "List of Recommended Symbols for Soil Mechanics" compiled and adopted by the International Society of Soil Mechanics and Foundation Engineering. They will be referred to letter ballot of Committee D-18.

Subcommittee 1 on Surface and Subsurface Reconnaissance (A. I. Johnson, chairman) is collaborating with Subcommittee 2 in planning the three-session Symposium on Soil Investigation to be held in June, 1963, at the Annual Meeting of the Society.

Subcommittee 2 on Sampling and Related Field Testing for Soil Investigation (J. P. Gnaedinger, chairman).—The subcommittee is working closely with the International Group on Soil Sampling and is collaborating with the Soil Mechanics Division of ASCE to avoid duplication of activities insofar as is possible.

The Subcommittee is also working closely with the Canadian Standards Assn. in the standardization of many items of mutual interest.

The following sections were established: (1) Section A on Nuclear Methods (A. I. Johnson, chairman), (2) Section B on In-Place Vane Shear Testing of Soils (E. B. Hall, chairman), (3) Section C on Diamond Core Drilling (George Sowers, chairman), (4) Section D on Auger Sampling of Soils (Rockwell Smith, chairman), and (5) Section E on Undisturbed Sampling and Penetration Tests of Soils (E. J. Zegarra, chairman).

The subcommittee recommended revisions of Methods D 1452, D 1586, and D 1587 as noted earlier in this report.

Subcommittees 2 and 8 are collaborating in a questionnaire regarding nuclear methods, the results of which will be reported upon at the 1963 Annual Meeting.

The subcommittee is also considering a proposed method for in-place vane shear testing of soils.

Subcommittee 3 on Texture, Plasticity, and Density Characteristics of Soils (E. E. Bauer, chairman) prepared the new tentative Method for Calibration of Mechanical Laboratory Soil Compactors; and proposed methods for Laboratory Determination of Moisture Content of Soil, and for Wet Preparation of Soil Samples for Grain-Size Analysis and Determination of Soil Constants. It also recommended that Method D 1558 be adopted as standard.

Under consideration are revisions of Methods D 698 and D 1557 to provide (1) tolerances on the volumes of the molds, (2) tolerances on weights and heights of drop of the rammers, (3) use of mechanical compactors, (4) drawings for the manual rammers and guide sleeves, (5) use of sector-faced rammers, and (6) use of a concrete cylinder as the base for the mold during compaction of the soil with the manual method.

Also under consideration is a proposed Method of Test for Relative Density of Cohesionless Soils.

Subcommittee 5, Structural Properties of Soils (G. F. Sowers, chairman) prepared the new Tentative Method of Test for Unconfined Compressive Strength of Cohesive Soils, appended to this report. It is currently considering proposed methods for other tests including direct shear, consolidation, and triaxial compression tests.

Subcommittee 6 on Physico-Chemical Properties of Soils (R. L. Handy, chairman).—The entire Committee D-18 membership was saddened by the passing of Donald T. Davidson, for many years chairman of Subcommittee 6. His responsibilities as chairman have been taken over by Mr. R. L. Handy.

Subcommittee 8 on Special and Construction Control Tests (B. S. Coffman, chairman) held a meeting during the 1962 Annual Meeting with all sections active.

Section A, Base Course and Subgrades for Highways and Airports, is working on the sand equivalent test method for presentation to the committee.

Section B, Earth Dams and Embankments, prepared the new Method of Test for Density in Place by the Rubber Balloon Method appended to this report.

A task group on nuclear surface methods for this determination is active.

Section C-1, Cement Stabilization, has proposed the adoption of Methods D 1632, D 1633, D 1634, and D 1635 as standard.

Section C-2, Bituminous Stabilization, developed a proposed recommended practice for soil-bituminous stabilized base course and is considering other methods.

Section C-4, Other Admixtures, is developing a method on the stabilizations of soils with lime.

Subcommittee 9 on Soil Dynamics (R. K. Bernhard, chairman) has recommended that the Tentative Definitions of Terms Relating to Soil Dynamics (D 1707 T) be continued as tentative without revision.

Reorganization of the subcommittee is being considered.

Subcommittee 10 on Bearing Tests of Soil in Place (P. P. Brown, chairman) recommended editorial changes in Methods D 1195 and D 1196 as noted earlier in this report.

Subcommittee 11 on Pile Load Bearing Tests (W. S. Housel, chairman) recommends continuation of Method D 1143 as tentative without revision. The subcommittee is also considering possible methods for lateral load tests on piles.

This report has been submitted to letter ballot of the committee, which consists of 118 voting members, 74 members returned their ballots, of whom 73 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. G. HOLTZ,
Chairman.

J. P. GNAEDINGER,
Secretary

REPORT OF COMMITTEE D-19
ON
INDUSTRIAL WATER*

Committee D-19 on Industrial Water held meetings on June 27 to 29, 1962, in New York, N. Y., and on Jan. 21 to 23, 1963, in West Palm Beach, Fla.

The Society published the papers included in the committee-sponsored Session on Industrial Water and Industrial Waste Water, presented on June 28, 1962, in New York, N. Y., as a Special Technical Publication (*STP No. 337*). Included are two of the papers of a similar session presented at the Fourth Pacific Area Meeting on Oct. 4, 1962, in Los Angeles, Calif.

In *Materials Research & Standards*, the following papers were published for the general interest of ASTM members: (1) "Gas Chromatographic Analysis of Aqueous Solutions," by R. A. Baker, Senior Staff Engineer, Franklin Institute, Philadelphia, in the December, 1962, issue; and (2) "The Political Puppet Called Purity," by J. E. Kinney, Consultant in Sanitary Engineering, Ann Arbor, Mich. in the January, 1963, issue.

The Third Printing of the Second Edition of the Manual on Industrial Water and Industrial Waste Water (*STP 148-F*) was published. Part I, the text chapters, was not revised; Part II, Compilation of Methods, was expanded to include all tentatives and standards accepted by and published by the Society through 1962.

The committee is proceeding with the revisions of the text chapters of its Manual, in preparation for the Third Edition, under the chairmanship of J. F. Wilkes. The revised Part I will include the following 15 chapters:

I. Water Sources and Supply,[a,c] S. K. Love, Water Resources Div., U. S. Geological Survey.
II. Uses of Industrial Water,[b] F. N. Kemmer, Nalco Chemical Co.
III. Effects of Composition on Water Use,[b,d] E. P. Partridge, Hagan Chemicals & Controls, Inc.
IV. Treatment of Process Water and Waste Water,[b,e] J. J. Maguire, Betz Laboratories, Inc.
V. Technology of Industrial Reuse of Water,[a] F. E. Clarke, Water Resources Div., U. S. Geological Survey.
VI. Pollution and Self-Purification of Streams,[b] R. S. Ingols, Georgia Inst. of Technology, Dept. of Applied Biology.
VII. Thermal Loading of Streams,[a] R. D. Hoak, Mellon Institute of Industrial Research.
VIII. Sampling and Flow Measurements of Industrial Water and Waste Water,[b] E. F. Davidson, Stone & Webster Engineering Corp.
IX. Subjective Examination of Water,[a] R. A. Baker, The Franklin Institute.
X. Analysis of Industrial Water and Industrial Waste Water,[b] J. H. Phillips, Nalco Chemical Co.
XI. Instrumental Monitoring of Water Quality.[a,f]
XII. Sampling and Instrumental Identification of Water-Formed Deposits,[b,c] C. E. Imhoff, Allis Chalmers Manufacturing Co.
XIII. Chemical Analysis of Water-Formed Deposits,[b] R. A. Larrick, Dearborn Chemical Co.

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

XIV. Radioactive Nuclides in Water,[b] Bernd Kahn, Robert A. Taft Sanitary Engineering Center.

XV. Water Technology for Nuclear Reactors,[a,g] W. J. Singley, Jr., Westinghouse Electric Corp.

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[a] One of six completely new chapters to be added to the Third Edition.

[b] One of nine existing chapters to be revised and updated in Third Edition.

[c] Includes material formerly in Chapter IV on "Composition and Classification of Industrial Water and Water-Formed Deposits"; Chapter I will contain material on composition and classification of industrial water, and Chapter XII will include classification of water-formed deposits.

[d] Will consider effects of water composition as it enters intakes. Effects of pollution on industrial uses will be retained.

[e] Desalination, considered as a treatment process for industrial water and waste water, covered in Chapter IV, will be covered, as well as discussions on pollution.

[f] Will be developed by author and associated sub-group; approach desired is automatic continuous monitoring or analysis for water quality surveillance.

[g] It was suggested that water uses in other critical processes requiring high-quality water also be covered in Chapter XV.

The report of activities of the Joint Committee on Uniformity of Methods of Water Examination submitted by F. E. Clarke and J. E. Kinney, ASTM representatives to JCUMWE, is summarized as follows:

JCUMWE approved official panel recommendations on a method for fluoride ion and circulated a method on electrical conductivity for approval of official recommendations. Panel reports were received on methods for ammonia; calcium and magnesium; carbonate, bicarbonate, and carbon dioxide; residual chlorine; chromium; lead; nitrate; nitrite; odor; silica; turbidity; and uniformity of reagents. A new panel was established on radiological procedures.

C. E. Imhoff was appointed as Results Adviser of the committee, effective January 1, 1963.

The committee at the Jan. 23, 1963, meeting approved the recommendations from Subcommittee I, Advisory, on the following: (1) the establishment of the Task Group on Planning under the jurisdiction of Subcommittee I, Advisory, and its scope, "To consider the application of the scope of Committee D-19 on Industrial Water to national needs and to recommend future programs of the committee and appropriate organization to carry out those programs." The membership of the task group include: R. C. Adams, F. E. Clarke, R. D. Hoak, J. E. Kinney, J. F. Wilkes, S. K. Love (ex-officio), and Max Hecht, acting chairman, and (2) the establishment of Subcommittee X on Significance of Water Composition, with a scope "To prepare interpretations of water characteristics for water resources application." R. C. Adams was appointed subcommittee chairman effective July 1, 1963, and J. K. Rice temporary vice-chairman.

The 1963 Max Hecht Award Certificate and Citation will be presented to J. F. J. Thomas, Head, Industrial Water Section, Mines Branch, Department of Mines and Technical Surveys, Ottawa, Canada, on June 26, 1963.

The committee records with sorrow the death of George A. Perley on Oct. 14, 1962.

The committee comprises 160 members. It operates with one advisory and nine technical subcommittees, and 85 task groups are distributed among the subcommittees. The committee's methods production is 76, of which 33 are published as tentative and 43 as Standard (exclusive of methods included and appended in this report).

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee D-19 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] All of the existing standards and tentatives referred to appear in their present form in the 1961 Book of ASTM Standards, Part 10 or its 1962 Supplement, except as indicated.

New Tentatives

as appended hereto.[2]

Methods of Test for:

Deuterium Oxide (Subcommittee II),
Deposit-Forming Impurities in Steam (Subcommittee VI),
Physical and Chemical Properties of Ion Exchange Resins (Subcommittee VI), and
Radioactive Cobalt in Industrial Water and Industrial Waste Water (Subcommittee IX).

Revisions of Tentatives

Methods of Test for Sulfate Ion in Industrial Water and Industrial Waste Water (D 516 – 59 T) (Subcommittee IV):

Section 11.—Change to read "Results by this method are precise to 1.0 per cent of the amount of sulfate ion present."

Section 20.—Change the beginning of the first sentence to read "The precision of this method depends on . . ." In the second sentence following "or 2 ppm, whichever is greater," insert a period and delete the rest of the sentence.

Section 31.—Change the last sentence to read "Single operator precision may be expected to be 0.5 ppm."

Section 24(b).—Change to read as follows:

(*b*) *Ion Exchange Column.*—A suitable continuous flow column may be prepared by pouring 30 cm of a washed, wet resin, 20- to 25-mesh size into a glass column of 9- to 10-mm ID and 50 ± 0.5 cm in length, the top of which widens to a reservoir of 50- to 55-mm ID and 10 ± 0.5 cm in length.

The resin is held on a suitable screen or filter plug and the flow is controlled by a length of 2-mm bore capillary tubing joined to the bottom of the column. This tubing is bent into a U shape and rises to about 13 mm above the resin bed and then makes a U bend downward for about 5 to 7 cm.

In the third line of the second paragraph, change "1 in. of washed resin" to read "25 mm of washed resin."

Tentative Methods of Test for Copper in Industrial Water and Industrial Waste Water (D 1688 – 62 T) (Subcommittee IV)

Section 31(a).—Change to read as follows:

31. (*a*) *Acetate-Phthalate Buffer.*—Prepare an acetate solution by dissolving 100 g of sodium acetate trihydrate ($NaC_2H_3O_2 \cdot 3H_2O$) in water and diluting solution to 500 ml with water. Make up a phthalate solution by dissolving 50 g of potassium acid phthalate ($HOOC \cdot C_6H_4COOK$) in 230 ml water heated to a temperature of about 80 C. While the phthalate solution is still warm, add sufficient phthalate solution to the acetate solution to produce a buffer having a pH of 5.4 as measured with a pH meter. Usually about 150 ml of the phthalate solution is required.

Section 35.—Change to read as follows:

35. The single operator and over-all precisions of the method can be expressed as follows:

$$S_T = 0.044X + 0.008, \quad \text{and}$$

$$S_o = 0.005X + 0.006$$

where:
S_0 = single operator precision in ppm,
S_T = over-all precision in ppm, and
X = concentration of copper in sample in ppm.

Adoption of Tentative as Standard Without Revision

Tentative Method of Test for Hexane-Extractable Matter in Industrial Waste Water (**D 1891 – 61 T**) (Sub-**committee VII**).

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[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[2] The new tentatives appear in the 1963 Supplement to *Book of ASTM Standards*, Part 10.

Adoption of Tentatives as Standard With Revision

Tentative Method of Test for Odor of Industrial Water and Industrial Waste Water (**D 1292 – 61 T**) (Subcommittee VII):

Revise as appended hereto.[3]

Revision of Standard and Reversion to Tentative

Standard Method of Test for Manganese in Industrial Water and Industrial Waste Water (D 858 – 49) (Subcommittee IV):

Revise and revert to tentative as appended hereto.[3]

Standard Method of Test for Nitrite Ion in Industrial Water (D 1254 – 60) (Subcommittee IV):

Revise and revert to tentative as appended hereto.[3]

Revision of Standard, Immediate Adoption

(requires nine-tenths affirmative vote)

Standard Method of Reporting Results of Analysis of Industrial Water and Industrial Waste Water (D 596 – 58) (Subcommittee II):

New Section 9.—Add a new Section 9 entitled "Odor" to read as follows, renumbering present Sections 9 and 10 as 10 and 11:

9. (*a*) A procedure for examination of industrial water for odor is given in the Method of Test for Odor in Industrial Water and Industrial Waste Water (ASTM Designation: D 1292).

(*b*) Report the odor intensity as odor intensity index calculated as follows:

$$\text{Odor intensity index} = 3.3 \log \left(\frac{200}{A}\right) + 3D$$

where:

A = milliliters of sample or milliliters of aliquot of the primary dilution used, and

D = number of 25:175 primary dilutions required to reach the determinable magnitude of odor intensity.

(*c*) The odor intensity may be calculated as threshold odor number if desired by the procedure described in Method D 1292, Appendix II.

Section 10(b).—Change to read as follows:

(*b*) The following table lists factors for interconversion of parts per million and equivalents per million of the common ions.[4]

Standard Definitions of Terms Relating to Industrial Water and Industrial Waste Water (D 1129 – 62) (Subcommittee II):

Revised Definition.—Change the present definition of "Basicity" to read "*Basicity.*—See Alkalinity."

New Definitions.—Add the following new definitions in their proper alphabetical sequence:

Anion-Exchange Material.—An ion-exchange material capable of the reversible exchange of negatively charged ions.

Atom Fraction of an Isotope.—The number of atoms of the isotope divided by the total number of atoms of that element in the sample. This ratio is analogous to mol fraction. For example, using the isotopes of oxygen, the atom fraction of O^{18} is

$$\frac{O^{18}}{O^{16} + O^{17} + O^{18}}$$

Atom Per Cent of an Isotope.—The atom fraction of an isotope expressed in per cent.

Cation-Exchange Material.—An ion-exchange material capable of the reversible exchange of positively charged ions.

Ion-Exchange Resin.—A synthetic organic ion-exchange material.

Mixed Bed.—A physical mixture of anion-exchange material and cation exchange material.

Normal Abundance of an Isotope.—The abundance in atom per cent of the isotopes of an element as it is found in nature.

Tentatives Continued Without Revision

Tentative Specifications for:

Equipment for Sampling of Water and Steam (**D 1192 – 51 T**) (Subcommittee III).

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[3] The revised tentatives and standard appear in the 1963 Supplement to *Book of ASTM Standards*, Part 10.

[4] The table is not included in this report but appears in the revised standard; see 1963 Supplement to *Book of ASTM Standards*, Part 10.

Tentative Methods of:

Sampling of Steam (**D 1066 – 59 T**) (Subcommittee III),
Sampling of Homogeneous Industrial Waste Water (**D 1496 – 57 T**) (Subcommittee III), and
Measurement of Beta Particle Radioactivity of Industrial Water and Industrial Waste Water (**D 1890 – 61 T**) (Subcommittee IX).

Tentative Methods of Test for:

Hydrazine in Industrial Water (**D 1385 – 58 T**) (Subcommittee IV),
Acidity and Alkalinity of Industrial Water (**D 1884 – 61 T**) (Subcommittee IV),
Dissolved Oxygen in Industrial Water (**D 888 – 49 T**) (Subcommittee IV),
Nickel in Industrial Water and Industrial Waste Water (**D 1886 – 61 T**) (Subcommittee IV),
Silica in Industrial Water (**D 859 – 55 T**) (Subcommittee IV),
Silica in High Purity Water (**D 1689 – 61 T**) (Subcommittee IV),
Suspended and Dissolved Solids in Industrial Water (**D 1888 – 61 T**) (Subcommittee IV),
Zinc in Industrial Water and Industrial Waste Water (**D 1691 – 59 T**) (Subcommittee IV),
Electrical Conductivity of Industrial Water and Industrial Waste Water (**D 1125 – 59 T**) (Subcommittee V),
Turbidity of Industrial Water and Industrial Waste Water (**D 1889 – 61 T**) (Subcommitee V), and
Effects of Water on Heat Exchange Materials (**D 1591 – 58 T**) (Subcommittee VI).

This report has been submitted to letter ballot of the committee, which consists of 160 members; 121 members returned their ballots, of whom 109 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

MAX HECHT,
Chairman.

ORRIN M. ELLIOTT,
Secretary.

REPORT OF COMMITTEE D-20
ON
PLASTICS*

Committee D-20 on Plastics has held three meetings during the past year: in New York, N.Y. on June 27 to 29, 1962, at the Annual Meeting of the Society; in Boston, Mass., Oct. 23 to 25, 1962; and in Cleveland, Ohio, Feb. 13 to 15, 1963.

The committee consists of 541 individuals, of whom 268 are voting members; 137 of the voting members are classified as producers, 60 as consumers, and 71 as general interest members.

The Boston meeting featured a 25th Anniversary Dinner with G. M. Kline and Robert Burns, two of the original members, presenting a two-part history of the committee. As part of a continuing series of indoctrination lectures sponsored by Subcommittee XIII on Statistical Techniques, E. C. Harrington presented a paper entitled "Sequential Experimentation in Factorial Space." A. G. H. Dietz also showed three-dimensional color slides of a recent trip around the world.

At the Cleveland meeting there was presented a series of three papers on "Armed Forces Requirements for Plastics Test Methods." S. P. Sotir represented the Army; H. W. Gilbert, the Navy; and T. J. Reinhart, Jr., the Air Force.

William A. Evans, an Honorary Member of Committee D-20, received an Award of Merit from the Society at the 1962 Annual Meeting.

The D-11/D-20 Coordinating Committee met on June 26, 1962, during the Annual Meeting. It was decided that revision of Committee D-20's definitions of "rubber" and "elastomer" would await action by Committee D-11. It was also suggested that Committee D-11 should forward to Committee D-20 for review insulated wire specifications where plastics are used for covering.

Representatives of Committees D-20 and C-16 met during the Conference of Technical Committee Officers on Nov. 20, 1962, to continue their coordination efforts on specifications and test methods of common interest.

The chairman of Subcommittee XXI on U.S.A. National Committee ISO/TC 61 will circulate new ISO documents to the pertinent subcommittee chairmen. Subcommittee VII on Analytical Methods will review several current ISO documents for which there is no present Committee D-20 standard.

Two new sections were formed due to industry demand: Section XVIII-K on Filament Winding Test Methods and Section XVII-F on Heat Joining.

Questionnaires circulated to members confirmed that Committee D-20 should not be concerned at present with monomers or with plastics for masonry.

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1962 Annual Meeting, Committee D-20 presented to the Society through the Administrative Committee on Standards the recommendations listed below which were accepted by the Administrative Committee on the dates indicated:

New Tentative Specifications for:

Rigid Poly(Vinyl Chloride-Vinyl Acetate) Compounds (D 2114 – 62 T) (Approved July 23, 1962),
Rigid Poly(Vinyl Chloride-Vinyl Acetate) Plastic Sheet (D 2123 – 62 T) (Approved September 11, 1962),
Cellular Polystyrene (D 2125 – 62 T) (Approved October 17, 1962),
Acetal Resin Injection Molding and Extrusion Materials (D 2133 – 62 T) (Approved October 17, 1962),
FEP-Fluorocarbon Molding and Extrusion Materials (D 2116 – 62 T) (Approved October 24, 1962), and
Propylene Molding and Extrusion Materials (D 2146 – 63 T) (Approved March 21, 1963).

New Tentative Methods of Test for:

Infrared Spectrophotometric Analysis of Components in Poly(Vinyl Chloride) Compounds (D 2124 – 62 T) (Approved September 11, 1962),
Determining Dimensions of Thermoplastic Pipe (D 2122 – 62 T) (Approved October 17, 1962),
Resistance of Rigid Cellular Plastics to Simulated Service Conditions (D 2126 – 62 T) (Approved October 17, 1962),
Water Absorption of Rigid Cellular Plastics (D 2127 – 62 T) (Approved October 17, 1962),
Softening of Organic Coatings by Plastic Compositions (D 2134 – 62 T) (Approved October 17, 1962),
Melting Point of Semicrystalline Polymers (D 2117 – 62 T) (Approved October 24, 1962),
Adhesion Ratio of Polyethylene Film (D 2141 – 63 T) (Approved March 21, 1963), and
Cyclic Pressure Strength of Reinforced, Thermosetting Plastics Pipe (D 2143 – 63 T) (Approved March 21, 1963).

New Tentative Recommended Practice for:

Oven Heat Stability of Poly(Vinyl Chloride) Compositions (D 2115 – 62 T) (Approved July 23, 1962).

Revision of Tentative Specification for:

Nylon Injection Molding and Extrusion Materials (D 789 – 62 T) (Approved September 11, 1962),
IPS Cellulose Acetate Butyrate Pipe (D 1503 – 62 T) (Approved October 17, 1962),
Rigid Acrylonitrile-Butadiene-Styrene (ABS) Plastics (D 1788 – 62 T) (Approved October 17, 1962),
TFE-Fluorocarbon Resin Molding and Extrusion Materials (D 1457 – 62 T) (Approved October 24, 1962),
Cast Methacrylate Plastic Sheets, Rods, Tubes, and Shapes (D 702 – 63 T) (Approved March 21, 1963), and
Methacrylate Molding and Extrusion Compounds (D 788 – 63 T) (Approved March 21, 1963).

Revision of Tentative Methods of Test for:

Short-Time Rupture Strength of Plastic Pipe, Tubing, and Fittings (D 1599 – 62 T) (Approved October 24, 1962),
Impact Resistance of Polyethylene Film by the Free Falling Dart Method (D 1709 – 62 T) (Approved October 24, 1962), and
Density of Plastics by the Density-Gradient Technique (D 1505 – 63 T) (Approved March 21, 1963).

Revision of Tentative Nomenclature Relating to:

Plastics (D 883 – 63 T) (Approved October 17, 1962, and March 21, 1963).

All of the new and revised standards referred to above appear in the 1962 Supplement to Book of ASTM Standards, Part 9, with the exception of those approved in March, 1963, which are available as separate reprints.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee D-20 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] All of the existing tentatives and standards referred to appear in their present form in the 1961 Book of ASTM Standards, Part 9.

New Tentative

Tentative Recommended Practice for Statistical Design in Interlaboratory Testing of Plastics, as appended hereto.[2]

Revision of Tentatives

Tentative Specification for Polyethylene Molding and Extrusion Materials (**D 1248 – 60 T**):

Section 13(c).—Add the following sentence: "Test results that break outside the gage-marks after extensive cold-drawing need not be discarded unless the break occurs between the contact surfaces of a grip."

Tentative Specification for Polytetrafluoroethylene Rod (**D 1710 – 60 T**):

Section 8(b)(1).—Add the following to Item (*1*):

Machine test specimen with any lathe permitting the follower assembly (Fig. 2) to be fastened to the carriage. Make the follower assemblies and cutter in accordance with Fig. 3. The followers are used to support the rod during machining. Equivalent apparatus that will accomplish this purpose may be used.

Select a follower whose bore is closest to but smaller than the rod. Machine the rod 0.05 ± 0.025 mm (0.002 ± 0.001 in.) smaller than the bore of the selected follower.

Center the axis of the bore of the follower with the axis of the headstock of the lathe. Bolt the follower assembly to the carriage.

Fasten a 15.5–cm. (6-in.) length of rod in the headstock. Insert the rod into the follower 3.5 cm. ($1\frac{3}{8}$ in.) beyond the open section of the follower. Check to be sure that the rod rotates without binding. (Readjust the position of the follower assembly on the carriage if binding occurs.)

Set the cutter at the open section of the follower as close to *A* (Fig. 2) as possible and, in one continuous operation, machine the tension specimen in accordance with Fig. 1. Machine at a speed fast enough to prevent whipping of the rod. The nominal diameter of the test section of the tension specimen shall be 60 per cent of the original nominal diameter of the rod as received for test.

New Figures 2 and 3.—Add new Figs. 2 and 3 as shown in the accompanying Figs. 1 and 2.[3]

Additional editorial changes consisting of addition of metric units are not included in this report.[3]

Adoption of Tentatives as Standard Without Revision

Tentative Specifications for:

Cellulose Acetate Molding and Extrusion Compounds (**D 706 – 61 T**),
Cellulose Acetate Butyrate Molding and Extrusion Compounds (**D 707 – 61 T**),
Ethyl Cellulose Molding and Extrusion Compounds (**D 787 – 61 T**),
Methacrylate Molding and Extrusion Compounds (**D 788 – 63 T**), and
Biaxially Oriented Styrene Plastics Sheet (**D 1463 – 58 T**).

Tentative Methods of Test for:

Stiffness of Plastics by Means of a Cantilever Beam (**D 747 – 61 T**),
Coefficients of Friction of Plastic Film (**D 1894 – 61 T**),
Absorbed Gamma Radiation Dose in the Fricke Dosimeter (**D 1671 – 59 T**), (Jointly with Committee D-9),
Water Absorption of Plastics (**D 570 – 59a T**),
Particle Size (Sieve Analysis) of Plastic Materials (**D 1921 – 61 T**),
Measuring Flatness of Plastics Sheet or Tubing (**D 1604 – 58 T**), and
Apparent Density of Rigid Cellular Plastics (**D 1622 – 59 T**).

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[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[2] The new tentative appears in the *1964 Book of ASTM Standards*, Part 27.

[3] Figures 1 and 2 and the editorial changes are not included in this report but appear in the revised tentative; see *1964 Book of ASTM Standards*, Part 26.

Tentative Recommended Practices for:

Determining Resistance of Plastics to Fungi (**D 1924 – 61 T**), and
Injection Molding of Specimens of Thermoplastic Materials (**D 1130 – 50 T**).

Adoption of Tentative as Standard With Editorial Changes

Tentative Method of Test for Flammability of Rigid Plastics over 0.050 in. in Thickness (**D 635 – 56 T**).

The editorial changes are not included in the report; they consist of adding metric units.

Revision of Standards Immediate Adoption

(requires nine-tenths affirmative vote)

Standard Method of Test for Flexural Properties of Plastics (D 790 – 61):

Revise as appended hereto.[3a]

Standard Method of Test for Gas Transmission Rate of Plastic Sheeting (D 1434 – 58):

Revise as appended hereto.[3a]

Withdrawal of Tentative

Tentative Specifications for Vinyl Chloride Polymer and Copolymer Rigid Sheets (**D 708 – 53 T**).

Withdrawal of Standard

Standard Specification for Vinyl Chloride-Acetate Resin Molding Compounds (**D 728 – 50**).

Tentatives Continued Without Revision

The committee recommends the continuation of all other tentatives under the jurisdiction of the committee not otherwise mentioned in view of the fact that work is currently in process on revision thereof, or, in a few cases, insufficient experience has been had to justify adoption as standard.

American Standard

The following tentatives have been approved by the American Standards Assn. as American Standard:

Tentative Method of Test for Stiffness Properties of Nonrigid Plastics as a Function of Temperature by Means of a Torsion Test (**D 1043 – 61 T**; ASA K65.2-1962), and
Tentative Specification for Polyethylene Molding and Extrusion Materials (**D 1248 – 60 T**; ASA C59.40-1963).

It is recommended that all tentatives being adopted as standard be submitted to ASA for approval as American Standard.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Mechanical Properties (G. D. Patterson, Jr., chairman) is developing high-speed tensile, film impact, torsion pendulum, puncture-tear, coefficient of friction, and hardness methods.

Formal reports have been made of interlaboratory work on high-speed tension testing and on the effects of specimen preparation for conventional tension testing. Eleven existing methods are being studied for significant revisions. Five tentative methods have been recommended for adoption as standards. Section Q on Hardness has been reactivated while Section P on Stiffness has been made inactive. Interest is being sought in reactivating sections on Scratch Resistance and Biaxial Strength.

[3a] The revised standards appear in the *1964 Book of ASTM Standards*, Part 27.

The status of projects now in progress is as follows:

Nature of Action and Title	Responsible Section	Ballot Status[4]		
		Sect.	Sub.	Comm.
New Methods:				
High Speed Tensile Properties	A	1		
Torsion Pendulum	O		1	
Revisions:				
D 882, Tensile Properties of Thin Plastic Sheeting	A			1
D 256, Impact Resistance	B			1
D 671, Flex Stress (Fatigue)	C			1
D 1043, Stiffness Properties by Torsion Test	F		2	
D 790, Flex Properties	F			2
D 1004, D 1922, D 1938, Tear Tests	M			1
D 695, Compressive Properties	T			1
Research:				
High Speed Tensile Properties of Films (round robin)	A			
Impact Testing of Films (round robin)	B			
Friction Properties of Solid Plastics (round robin)	R			
Improved Terminology of Testing Speeds	A			
Tensile Impact of Solid Plastics	B			
Puncture Tear Testing of Films (round robin)	M			
Barcol Indentation Hardness (round robin)	Q			

Subcommittee II on Radiation Methods (Joint with Committee D-9) (D. J. Metz, chairman) is developing dosimetry methods for the various types of radiations:

Section A on Exposure Methods is currently attempting to ascertain the degree of use of Recommended Practice D 1672 before recommending its adoption as standard.

Section B on Gamma Ray Dosimetry is attempting to establish an interlaboratory test program on the oxalic acid high intensity gamma ray dosimeter.

Section C on Beta Particle and Accelerated Electron Dosimetry has been inactive due to a lack of a chairman. A new chairman has recently been appointed.

Section D on Neutron Dosimetry, has submitted to Subcommittee letter ballot the Method of Calculation of Neutron Dose to Organic Materials and Application of Threshold-Foil Measurements.

Section E on Gamma Ray Dosimetry in Reactor Irradiations is working on the carbon-wall ionization chamber method.

Section F on Correlation of Absorbed and Exposure Doses is developing analytical expression and curves for correlation of the gamma ray dose absorbed in the Fricke Dosimeter (D 1671) to the dose absorbed in an organic plastic of a given arbitrary composition.

The status of subcommittee projects now in progress is as follows:

Nature of Action and Title	Responsible Section	Ballot Status[4]		
		Sec.	Sub.	Comm.
Research:				
Oxalic Acid System for High Level Gamma Ray Dosimetry	B			
Carbon - Wall Ionization Chamber for Discriminating Ray Dose in Reactor Irradiations	E			
Correlation of Absorbed and Exposure Doses	F			
New Method:				
Calculation of Neutron Dose to Organic Materials and Application of Threshold Foil Measurements	D		3	

Subcommittee III on Thermal Properties (D. L. Engle, chairman) has continued its study of methods related to melting, flow, flammability, brittleness, and ablation testing of plastics. All sections have considered carefully ISO/TC 61 documents relating to their activities.

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[4] Explanation of ballot status:
1 = ballot authorized,
2 = ballot in progress, and
3 = ballot passed.

Standards that have completed Committee D-20 letter ballot or have been accepted by the Society during the year are listed elsewhere in this report.

Section A on Softening and Melting has completed work on melting point (D 2117) and is undertaking further improvement of the Vicat test (D 1525). ISO R-75 and D 648 were found to be compatible.

Section C on Flammability continues a high level of activity on a variety of flammability methods. The importance of these standards in various building codes provides a continuing challenge for creative work.

Section D on Flow is one of the best-attended sections of Committee D-20, and a wide range of rheological tests are under active consideration by four task groups.

Section L on Ablation is revising its scope to cover only flame "ablation" testing, and has transferred interest in plasma arc methods to the new Committee E-21.

The status of projects now in progress is as follows:

Nature of Action and Title	Responsible Section	Ballot Status[4]		
		Sect.	Sub.	Comm.
New Method:				
Conversion of NEMA LP1-7.07 to ASTM standard	C	1		
Revisions:				
D 621, to describe jig for composite specimens	A		1	
D 746, to add appendix on calibration from Committee D-9	H		1	
D 757, for modernization	C	1		
D 1525, to change heating rate from 50 to 120 C per hr	A		1	
D 1238, major revision	D	1		
Research:				
Vicat test (D 1525)	A			
Oxy-acetylene torch ablation test on panels (round robin)	L			
High shear rheological tests	D			
Flow rate ratios—validity and methods	D			

Subcommittee IV on Optical Properties (J. H. Ross, Jr., chairman).—Section C has initiated a new interlaboratory test on gloss measurement of plastic film. The first interlaboratory test showed significant differences due to methods of sample handling. Section E has concluded that when the conditions are specified for ranking the yellowness of translucent specimens by transmitted light, visual ranking correlates with instrumental measurements. Therefore, the Method of Test for Yellowness Index of Transparent and Opaque Plastics (D 1925) has been revised to include translucent specimens, and this revision will be balloted.

The status of projects now in progress is as follows:

Nature of Action and Title	Responsible Section	Ballot Status[4]		
		Sect.	Sub.	Comm.
Revision:				
D 1925, Measurement of Yellowness of Translucent Plastics	E		2	2
Research:				
Measurement of Gloss of Plastic Films (round robin)	C			

SUBCOMMITTEE V.—PERMANENCE

(J. B. DeCoste, chairman).

Nature of Action and Title	Responsible Section	Ballot Status[4]		
		Sect.	Sub.	Comm.
Adoption as Standard:				
D 570, Water Absorption of Plastics	...			1
D 1499, Operation of Light and Water Exposure Apparatus	E			1
D 1924, Resistance of Plastics to Fungi	M			1
Revision:				
D 1435, Outdoor Weathering of Plastics	H			
New Methods:				
Effect of Fluorescent Light (round robin)	A			

Nature of Action and Title	Responsible Section	Ballot Status Sect.	Ballot Status Sub.	Ballot Status Comm.
Exposure of Plastics at Elevated Temperatures (above 300 C)	H	2	2	
Staining of PVC Compositions by Rubber Compounding Ingredients	K			3
General Staining Test for Plastics	K	1	1	
Resistance of Plastics to Bacteria	M			
Research:				
Yellow-Dyed Acrylic Chip	A			
Xenon Arc Artificial Weathering (round robin)	E			
Environmental Stress Cracking by Flexural and Tensile Methods	N			
Papers Presented:				
Allen, E., "A Monitoring Device for Ultraviolet Energy in the Atlas Xenon Weatherometer."	A			
Kamal, M. R., "Preliminary Fundamental Studies on the Weathering of Plastic Materials."	H			
Klausmeier, R. E., "Effect of Various Bacteria on Plasticizers."	M			

Subcommittee VII on Analytical Methods (J. N. Jessup, chairman) is developing physical, spectrophotometric, and gas chromatographic methods.

Section A on Physical Methods is revising Methods D 792 and D 1895, and is presently considering writing standards for four ISO Recommendations which fall under the jurisdiction of this subcommittee.

Section B on Extraction Methods has reviewed the areas of interest and found that insufficient interest exists at the present time in extraction methods. However, the subcommittee is reviewing more specific extraction problems and will reactivate at a time when the subcommittee feels work is necessary.

Section C on Gas Chromatography has reviewed many methods with respect to plastics. A task group is now actively working on methods for the analysis of thermoplastics and vinyl chloride monomer.

Section G on Gas Permeability has revised the present method (D 1434) for gas transmission rate. A task group has been organized to develop an alternate method utilizing apparatus and techniques which will allow determinations over broader temperature and pressure ranges, thus broadening the evaluation of various films.

Section H on Spectroscopic Methods has completed the writing of a new method for the analysis, by infrared methods, of poly(vinyl chloride) compounds. Another new method on the analysis of methyl groups in polyethylene is to be written shortly. A task group has also been organized to study and develop a method for the measurement of unsaturation in polyethylene.

The status of projects now in progress is as follows:

Nature of Action and Title	Responsible Section	Ballot Status[4] Sect.	Ballot Status[4] Sub.	Ballot Status[4] Comm.
Revisions:				
D 1505, Density by Density Gradient Technique	A			3
D 1434, Gas Transmission Rate of Plastic Sheeting	G			3
New Method:				
D 2124, Infrared Spectrophotometric Analysis of Components in Poly(Vinyl Chloride) Compounds	H			3

Subcommittee IX Molds and Molding (K. A. Weston, chairman).—Section A on Compression Molded Test Specimens has prepared a new Recommended Practice for Compression Molding Styrene-Butadiene Test Specimens which is now ready for Committee D-20 ballot. A request has been received from Section XV-N for a method of preparing ABS compression molded test specimens.

Section H on Injection Processes has conducted an interlaboratory test to determine the consistency of physical data obtained from injection molded styrene-butadiene test specimens. The data obtained showed an undesirable spread between 3- and 8-oz capacity molding machines; therefore, another interlaboratory test will be conducted which will include at least two machines of each capacity.

Section K on Extrusion Processes has organized an interlaboratory test to determine the correlation of data from physical tests on compression molded and extruded test specimens. If consistent data are obtained, a method will be prepared for predicting the characteristics of extruded plastics from compression molded specimens.

The status of projects now in progress is as follows:

Nature of Action and Title	Responsible Section	Ballot Status[4]		
		Sect.	Sub.	Comm.
New Method:				
Compression Molding Styrene-Butadiene Test Specimens	A			1
Research:				
Test Specimen for Extrusion Grades of Plastics (round robin)	K			
Molding Injection Styrene-Butadiene Test Specimens (round robin)	H			

Subcommittee X on Definitions (B. G. Achhammer, chairman) is continuing work on the development of definitions relating to plastics and of abbreviations for plastics terms. "The Word List for Glossary of ASTM Definitions" prepared by Committee E-8 on Nomenclature and Definitions is being reviewed to determine whether the definitions under the jurisdiction of Committee D-20 are in conflict with definitions of other ASTM Committees. The Manufacturing Chemists' Assn.-Society of Plastics Industry (MCA-SPI) Joint Committee on Identification of Plastics has indicated an intention to use Abbreviations D 1600.

The status of projects now in progress is as follows:

Nature of Action	Ballot Status[4]	
	Sub.	Comm.
New Definitions for:		
Compound, n		3
Dry-Blend, n		3
Fiber-Show		3
Powder Blend—See Dry-Blend		3
Sun Hour—Add to terms not recommended		3
Ultraviolet Sun Hour—Add to terms not recommended		3
Acetal Plastics, n	1	
Propylene Plastics, n	1	
Fluorocarbon Resins, n	1	
Withdrawal of Definitions:		
Weathering (from D 883)		3
Definitions under "Terms Not Recommended" (from D 883)		3
Revised Definitions:		
Nonrigid, Semirigid, and Rigid Plastics		1
Ethylene, Styrene, Vinyls, etc. to correspond to Propylene Plastics	1	
Recommendations:		
Retain Abbreviations D 1600 as tentative	3	

Subcommittee XI on Editorial Review (G. M. Kline, chairman) has reviewed all proposed new and revised specifications, methods of test, and recommended practices, and has submitted recommendations for editorial revision.

Subcommittee XII on Polyolefin Plastics (J. B. Howard, chairman) prepared the new Specification for Polypropylene Molding and Extrusion Materials (D 2146) accepted by the Standards Committee. This is the second "cell-type" specification in Committee D-20 area.

The status of projects now in process is as follows:

Nature of Action and Title	Responsible Section	Ballot Status[4]		
		Sec.	Sub.	Comm.
New Method:				
Stability of Polypropylene	B	1		
Revisions:				
D 1248, Polyethylene Molding and Extrusion Materials:	A			
Conversion to "cell-type" format				
Use of "nominal" in Density section			2	
Use of "max" and "min" for densities				
Allowance for jaw breaks				2
D 1693, Environmental Stress-Cracking of Type I Ethylene Plastics	C			
D 1928, Preparation of Compression - Molded Polyethylene Test Samples	D			
Research:				
Tensile Impact Testing of Polypropylene	B			
Constant Load Stress-Rupture Test for Polyethylene	C	1		
Apparent Viscosity of Polyolefin Plastics by Torque-Measuring Plastometer	C			

Subcommittee XIII on Statistical Techniques (R. H. Supnik, chairman).—Section B on Experimental Design prepared the new Recommended Practice for Statistical Design of Interlaboratory Testing of Plastics as appended to this report. Five unresolved negative votes were based on the feeling that this practice belongs under the jurisdiction of Committee E-11 on Quality Control of Materials. It was voted to submit the practice with the comment that Committee D-20 would like a Committee E-11 practice that can be used by all committees. The current "Manual on Method for Conducting an Interlaboratory Study of a Test Method" (*ASTM STP 335*) is not adequate for the needs of Committee D-20 at the present time.

Subcommittee XV on Thermoplastic Materials (W. D. Harris, chairman).—Section D has been conducting interlaboratory tests and preparing methods to test blown bottles for impact strength, permeability, environmental stress crack resistance, and column crush strength.

Having completed its work on methyl methacrylate plastics, Section B has been placed on inactive status.

Section Q on Polycarbonate Resins has solved some very difficult problems in the past two years and now has a proposed specification on polycarbonates which should be ready for section ballot in the latter part of this year.

The subcommittee recommended the adoption as standard of Specifications D 706, D 707, D 787, D 788, and D 1710; and the continuation as tentative of D 703, D 1430, D 1755, D 1784, D 1823, D 1824, and D 1892 since revisions are pending.

The status of projects now in process is as follows:

Nature of Action and Title	Responsible Section	Ballot Status[4]		
		Sec.	Sub.	Comm.
New Methods:				
Environmental Stress Crack Resistance of Blown Bottles	D	2		
Impact Resistance of Blown Bottles (round robin)	D			
Permeability of Blown Bottles (round robin)	D			
Column Crush Strength of Blown Bottles (round robin)	D			
Revisions:				
D 1784, Rigid Poly(Vinyl Chloride) Compounds	A	2		
D 1432, Nonrigid Vinyl Chloride Polymer Compounds	A			
D 1430, Polychlorotrifluoroethylene Molding and Extrusion Materials	L			
D 1788, ABS Plastics	N	1		

Subcommittee XVI on Thermosetting Materials (H. W. Kuhlmann, chairman) is developing specifications and test methods for thermosetting plastic materials.

Section A on Phenolic Materials is revising Specification D 700.

Section D on Allyl Materials is currently studying revisions of Specification D 1636.

Section E on Epoxy Materials is currently reviewing the status of Specification D 1763, and a task group is considering a specification for epoxy molding compounds.

Section G on Plastics for Tooling is actively engaged in three areas relative to developing test methods for plastics tooling compounds: (1) viscosity methods, possibly modifying Method D 1824 to make it usable for tooling resins, (2) gel time tests in which various commercially available instruments are being considered, and (3) general physical and mechanical properties tests.

The status of projects now in process is as follows:

Nature of Action and Title	Responsible Section	Ballot Status[4]		
		Sect.	Sub.	Comm.
Revisions:				
D 700, Phenolic Molding Compounds	A			2
D 1636, Allyl Molding Compounds	D			
Research:				
Specification for Epoxy Molding Compounds	E			
Methods for Plastics Tooling Compounds	G			

Subcommittee XVII on Thermoplastics Pipe and Fittings (Joint SPI-ASTM) (Frank W. Reinhart, chairman) is developing specifications and test methods for plastic pipe, fittings, and cements. Recommended practices for installation and joining are also under consideration. A method to determine hydrostatic design stresses is being studied by interlaboratory testing. A new Section F on Heat Joining was formed this year. Establishment of USANC on ISO TC 5/SC6 was also accomplished. Work was initiated to develop a polyethylene drain, waste, and vent pipe specification; sewer and drain pipe specifications; and an electrical conduit specification. The problems of test methods for water hammer and torque strength are also under study.

The status of projects now in process is as follows:

Nature of Action and Title	Responsible Section	Ballot Status[4]		
		Sect.	Sub.	Comm.
New Specifications:				
Polyethylene Plastic Pipe (SDR-PR)	C			3
Poly (Vinyl Chloride) (PVC) Plastic Pipe (SDR-PR)	A			3
Acrylonitrile - Butadiene - Styrene (ABS) Plastic Pipe (SDR-PR)	D			3
Solvent Cement for ABS Plastic Pipe and Fittings	E			3
Insert Fittings	J			
ABS Fittings, Threaded	I		3	
ABS Fittings, Socket	I		2	
PVC Fittings, Threaded	H		2	
PVC Fittings, Socket	H		2	
ABS Drain, Waste & Vent Pipe and Fittings	K		2	
PVC Drain, Waste & Vent Pipe and Fittings	K		2	
Solvent Cement for PVC Pipe and Fittings	E		2	
Withdrawal:				
D 1528, ABS SWP Size Pipe	D			3
Revision of Specifications:				
D 1785, PVC Pipe	A		2	
D 2104, PE Pipe	C		2	
D 1527, ABS Pipe	D		2	
New Methods:				
Extrusion Quality of PVC Pipe by Acetone Immersion	G			3
Impact Resistance of Plastic Pipe and Fittings	G		2	
Parallel-Plate Bearing Test	G		2	
Use of ISO Stress Formula	G			3
Measurement of Dimensions of Plastic Pipe	G			3
Revision of Methods:				
D 1598, a general revision	G			3
addition for testing with gases	G			1
D 1599	G			3
New Recommended Practice:				
Installation Procedure for Sewer and Drain Pipe	K		3	

All these activities are coordinated closely with those of the Plastics Pipe Inst. (successor to the Thermoplastic Pipe Division of SPI), a division of the Society of Plastics Industry.

Subcommittee XVIII on Reinforced Plastics (G. R. Rugger, chairman) is active in preparing specifications, test methods, and recommended practices for reinforced plastics.

Section B on Reinforced Pipes and Fittings is a Joint ASTM-SPI section. Test methods to determine burst characteristics have been prepared. Efforts are now being made to assign pressure ratings for pipe. As agreement is reached upon standards or pipes, more time and effort will be devoted to developing standards for fittings to be used with the pipe.

Section C on Corrugated Panels is also an ASTM-SPI joint section. Efforts to include requirements for panels utilizing 6-oz mats are currently under way.

Section E on Reinforcements is considering a revision of Specifications and Methods D 1529 since other constructions are now available. The preparation of a classification system for rovings and yarns is being considered.

Section G on Heat-Resistant Reinforced Materials has been spending its time developing test methods. These methods will be coordinated with other Subcommittees so that the final methods will be usable by all rather than having several similar methods on the books. Once the test methods are available, specifications for this type of material can be prepared.

Section H on Chemical-Resistance Test Methods is currently working on tests applicable to the chemical processing industry. This activity is being coordinated with the Corrosion-Resistant Structures Subcommittee of the Reinforced Plastics Division of SPI. Consideration is being given to writing test methods applicable to materials of interest to the Military.

Section I on Inspection and Nondestructive Test Methods has made progress on visual standards for reinforced plastics. Procedures for radiographic and ultrasonic techniques are in preparation.

SUBCOMMITTEE XVIII.—REINFORCED PLASTICS

Nature of Action and Title	Responsible Section	Ballot Status[4]		
		Sect.	Sub.	Comm.
New Specifications:				
Reinforced Plastic Pipe	B			3
Woven Roving Fabric for Polyester Laminates	E			3
Glass Fiber Rovings and Yarns	E	1		
New Methods and Practices:				
Pipe Crush Test	B	1		
Pipe Chemical Resistance	B	1		
Exposure of Plastics at Elevated Temperatures	G		1	
Physical Properties at Subnormal and Supernormal Temperatures	G		1	
Interlaminar Shear Strength	G		1	
Visual Defects of Glass Laminates	I		2	
Visual Defects of Transfer Molded Parts	I		2	
Reactivity of Premix Compounds	J	1		
Fabrication of Ring Test Specimens	K		1	
Horizontal Shear Strength by Short Beam	K		1	
Split Disk Tension Test	K	2		
Time to Failure of Plastic Pipe under Long Term Hydrostatic Pressure	B			3
Cyclic Pressure Strength of Reinforced Thermosetting Plastic Pipe	B		3	
Interlaminar Shear Specimens by Horizontal Ring Punch Out	K	1		
Determination of Interlaminar Shear by Ring Punch Out	K	2		
Doily Reinforcements for On-Axis Holes in Fiber Glass Filament Reinforced Plastic Pressure Vessels	K	2		
Revisions:				
D 1919, Corrugated Sheet	C			1
D 2105, Longitudinal Tensile Test for Reinforced Pipe	B			3
D 1694, Threads for Reinforced Thermosetting Pipe	B	1		

Section J on Premix and Prepreg Materials has been comparing results obtained from the different methods of determining flow of thermosetting materials. A test method for comparing the reactivities of premix is being circulated for comments.

Section K on Filament Winding Test Methods has been active in developing test methods which will be suitable. Difficulties have been encountered due to the fact that each laboratory has made slight modifications in supposedly standard techniques. In addition to the test methods, specimen preparation techniques have had to be prepared simultaneously.

SUBCOMMITTEE XIX.—PLASTIC FILM AND SHEETING

(K. A. Kaufmann, Chairman)

Nature of Action and Title	Responsible Section	Ballot Status[4]		
		Sec.	Sub.	Comm.
Adoption as Standard:				
D 1463, Spec. for Biaxially Oriented Styrene Plastics Sheet	E			3
D 1604, Measuring Flatness of Plastics Sheet or Tubing	B			3
Revision of Specifications:				
D 702 Spec. for Cast Methacrylate Plastic Sheets, Rods, Tubes, and Shapes	D			3
D 788, Spec. for Methacrylate Molding and Extrusion Compounds	D			3
Withdrawal of Specification:				
D 708, Vinyl Chloride Polymer and Copolymer Rigid Sheets	A			3
Revision of Tentative				
D 1709, Impact Resistance of Polyethylene Film by the Free Falling Dart Method	B			3
New Tentatives:				
D 2123, Spec. for Rigid Poly (Vinyl Chloride-Vinyl Acetate) Plastic Sheet	A			3
D 2141, Adhesion Ratio of Polyethylene Film	F			3
Proposed Specification:				
Cellulose Acetate Butyrate Film and Sheeting	C		1	
Specifications Under Consideration:				
Corrugated Poly (Vinyl Chloride) Sheeting	A			
Polypropylene Film and Sheeting	B			
Cellulose Propionate Film and Sheeting	C			

The status of projects now in process in Subcommittees XVIII and XIX are given on the preceding page.

Subcommittee XX on Cellular Plastics (R. E. Jones, chairman) continues in its development of methods of tests for physical properties of the final foam. Work is in progress on water absorption (revision of Method D 2127), porosity by the air pycnometer, volume by displacement, and thermal conductivity by the line heat source.

Also in progress is work on processibility tests for styrene and urethane foams. This work includes expandability, volatile content, and water content for the styrene beads, and rate of rise for the urethane foam.

The raw materials test section is initiating work on amine equivalent for modified isocyanates. Active cooperation with ASTM Committee C-16 continues in the development of specifications for cellular plastics used as thermal insulation.

The status of projects now in process is as follows:

Nature of Action and Title	Responsible Section	Ballot Status[4]		
		Sect.	Sub.	Comm.
New Tentatives:				
Specification for Rigid Urethane Foam	B		1	
Method for Rate of Rise Properties of Urethane Foam	A		1	
Research:				
Method for Thermal Conductivity of Cellular Plastics by Means of a Line Heat Source	A		1	

Subcommittee XXI, U.S.A. National Committee, ISO/TC 61 (A. C. Webber, chairman).—ISO/TC 61 held its 12th Annual Meeting in Warsaw, Poland, Sept. 17–22, 1962. The largest meeting to date comprised 131 delegates from 16 countries including 11 from the United States. The increased participation was due largely to pressure from the European Market Assns. and to increased activity in the preparation of specifications for materials. Since both European Market Assns. have agreed to use ISO standards wherever possible, their preparation becomes more urgent.

The leaders accepted in principle the proposal of the Secretariat that work on tests and specifications for plastics products should be undertaken only after considering each proposal on its own merit. In this connection, a proposal to begin work on vinyl asbestos floor tile was rejected. It was agreed to continue the present organization of Working Group 9 on specifications in which the work on each material is assigned to a task group which is discharged when the assignment is completed. Because of the number of specifications on which work must proceed simultaneously, some task groups of Working Group 9 will meet in the week preceding the Plenary Session of future meetings.

The meeting was highly productive with the 10 working groups holding 21 sessions and discussing about 60 items in the program of work. The following table summarizes the status of the work. A copy of the Annual Report for 1962, which lists all documents by name and status, may be obtained from the secretary of USANC, H. W. Dailey, at the American Standards Assn. office.

ISO Recommendations published	24
Draft ISO Recommendations accepted, awaiting Council approval	11
Draft ISO Recommendations being balloted	8
Draft Proposals being circulated to letter ballot of TC-61	17
Items being actively studied in Working Groups	34

The next meeting of TC-61 will be held in London, Sept. 8 to 14, 1963.

This report has been submitted to letter ballot of the committee, which consists of 319 voting members; 212 members returned their ballots, of whom 200 have voted affirmatively and 2 negatively.

Respectfully submitted on behalf of the committee,

J. E. HANNA,
Chairman.

C. F. FERRARO,
General Secretary.

REPORT OF COMMITTEE D-22
ON
ATMOSPHERIC SAMPLING AND ANALYSIS

Committee D-22 on Methods of Atmospheric Sampling and Analysis met on June 12 and 13, 1963, in Detroit, Mich. Total membership of the committee at the time of this meeting was 85, which consisted of 61 active members, 15 consulting members, and 9 company members that do not have individuals designated to represent them at this time.

Good progress has been reported on the Intersociety "Manual of Methods for Ambient Air Sampling and Analysis" sponsored by the following six societies: American Conference of Governmental Industrial Hygienists, American Industrial Hygiene Assn., Air Pollution Control Assn., American Public Health Assn., American Society of Mechanical Engineers, and ASTM. Moyer D. Thomas and Morris Katz represent ASTM on this intersociety project. Mr. Thomas has been selected to be editor of the manual.

During the year, the second edition of the compilation of *ASTM Standards on Methods of Atmospheric Sampling and Analysis* was published.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature and Units (E. R. Hendrickson, chairman) is continuing its work on the following items: (1) report on standard symbols and abbreviations for use in air pollution practice, (2) set of conversion factors for radioactive materials in the air, and (3) definitions of radioactive materials in the air.

Subcommittee II on Methods of Sampling and Analysis (H. M. Chapman, chairman) has prepared a draft of a proposed Tentative Method for Preparation of Aerosol Samples for Electron Microscopy.

A description of the fallout monitoring system used at the Health and Safety Laboratory of the New York Operation Office, U. S. Atomic Energy Commission, was submitted to the subcommittee and discussed by Robert Morse of the AEC.

A task group has been assigned to investigate the need for a suggested modification in the present Method of Test for Oxidant (Ozone) Content of the Atmosphere (D 1609).

The Task Group on Sampling for Particulate Matter has undertaken a new assignment—to prepare a method for collection of particulate matter by impaction techniques.

Subcommittee III on Instrumentation (M. D. Thomas, chairman) received again for reconsideration the proposed Method of Test for Continuous Measurement of Nitric Oxide, Nitrogen Dioxide, and Ozone in the Atmosphere, since voting was light on this proposal and there was a question as to whether it received the requisite majority. Further there was one negative vote which however, was changed. The vote now has been resolved and it is planned to resubmit the item to the Standards Committee for action.

The subcommittee is considering the

recommendation of minor changes in or additions to the Method of Test for Mass Concentration of Particulate Matter in the Atmosphere (Continuous-Measurement Light-Scattering Method) (D 1899) to accommodate the method to recent developments.

Subcommittee III is again studying a proposed Method for Dustfall According to Wind Direction and Velocity which is in draft form.

Progress has been made also on two proposed Methods of Test for Hydrogen Fluoride Concentration in the Atmosphere by (1) Continuous Recording of Fluorescent Quenching, and (2) Spectrophotometric Method.

Respectfully submitted on behalf of the committee,

M. D. THOMAS,
Chairman.

A. T. ROSSANO,
Secretary.

REPORT OF COMMITTEE D-23
ON
CELLULOSE AND CELLULOSE DERIVATIVES*

Committee D-23 on Cellulose and Cellulose Derivatives held one meeting during the year: in Atlantic City, N. J. on Sept. 12, 1962. The Executive Subcommittee also met in New York, N. Y. on Feb. 19, 1963.

The committee consists of 47 members, of whom 14 are classified as producers, 12 as consumers, and 21 as general interest members.

As a result of answers to a comprehensive questionnaire sent to Committee D-23 membership, the committee is considering the improving of some of their 16 present standards to take advantage of new instrumental techniques, and is also surveying possible new areas of work.

RECOMMENDATIONS FOR SOCIETY ACTION

Committee D-23 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] All of the existing tentatives referred to appear in their present form in the 1961 Book of ASTM Standards, Part 6.

Revision of Tentative

Tentative Methods of Testing Sodium Carboxymethylcellulose (D **1439** - **61 T**):

Section 1(b).—In the listing of test procedures add the following:

Sodium Chloride............. 36 to 41

Footnote 2.—Add "In 1963 there was added a potentiometric method for the determination of sodium chloride."

New Sections.—Add new Sections 36 to 41 entitled "Sodium Chloride" as appended hereto.[2]

Adoption of Tentatives as Standard Without Revision

Tentative Methods of Test for:

Chromatographic Analysis of Chemically Refined Cellulose (D **1915** - **61 T**), and

Carboxyl Content of Cellulose (D **1926** - **61 T**).

Adoption of Tentative as Standard With Editorial Change

Tentative Methods of Testing Cellulose Acetate (D **871** - **61 T**):

Section 13(b).—Insert the following sentence at the end of the paragraph: "Proceed in accordance with Paragraph (*d*)."

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] EDITOR'S NOTE.—No action was taken at the Annual Meeting on the Report of Committee D-23 in the absence of a quorum at the session when the report was scheduled for presentation. Alternate procedures were used in securing Society action on the recommendations, set forth herein, as follows:

The Administrative Committee on Standards accepted on Sept. 20, 1963, the revision of Tentative Methods D 1439 - 61 T.

Through the new interim procedure for standards, the Society accepted on Nov. 19, 1963, the three methods for adoption as standard.

[2] The new sections are not included in this report but appear in the revised tentative; see 1963 Supplement to *Book of ASTM Standards*, Part 6.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature and Definitions (Kyle Ward, Jr., chairman) is comparing terms in Definitions D 1695 with those defined by other ASTM committees and also in a preliminary thesaurus of pulp technology.

Subcommittee II on Cellulose (K. A. Kuiken, chairman) has organized task groups on optical methods for color of pulp and paper, and on lignin in pulp and paper.

Subcommittee III on Organic Esters (L. B. Genung, chairman) has recommended adoption of Methods D 871 as standard, with one minor editorial change.

Subcommittee V on Cellulose Ethers (R. W. Swinehart, chairman) has included a procedure for the potentiometric titration of chlorides in sodium carboxymethylcellulose in the revision of Method D 1439 as appended hereto.[2] A few revisions in Methods D 1347 are being made to bring the method up to date. Task groups on hydroxyethyl cellulose and methyl hydroxypropyl cellulose have been organized; methods are being prepared, and samples will be analyzed cooperatively.

This report has been submitted to letter ballot of the committee, which consists of 47 voting members; 32 have returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. K. WILSON,
Chairman.

W. W. BECKER,
Secretary.

REPORT OF COMMITTEE D-24
ON
CARBON BLACK*

Committee D-24 on Carbon Black and its subcommittees held two meetings during the year: in New York, N. Y., on June 26, 1962, and in Montreal, Quebec, on Feb. 5, 1963.

The committee consists of 43 members, of whom 9 are classified as producers, 25 as consumers, and 9 as general interest members.

In its eight years of existence, Committee D-24 has sponsored ten standard methods for testing carbon black, four tentative methods for sampling or testing carbon black and, jointly with Committee D-11 on Rubber and Rubber-Like Materials, a tentative specification for carbon black in natural rubber. Committee members expressed their appreciation to the past chairman, N. P. Bekema, U. S. Rubber Company.

RECOMMENDATION ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1962 Annual Meeting, Committee D-24, jointly with Committee D-11 on Rubber and Rubber-Like Materials, presented to the Society through the Administrative Committee on Standards revisions of the Tentative Specification for Carbon Blacks Used in Rubber Products (D 1765 – 61 T). The revision was accepted by the Standards Committee on December 17, 1962, and the revised specification is available as a separate reprint.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Chemical Tests (L. G. Mason, chairman) is considering methods for assessing carbon black structure or chain-like particle aggregation. Methods under investigation include the oil absorption of carbon black and the specific volume of carbon black under compression.

Subcommittee III on Optical Tests (J. E. Smith, chairman) is continuing work to improve the benzene discoloration method (D 1618) and recommends its continuation as tentative without revision.

Subcommittee V on Sampling (J. E. Smith, chairman) recommends the adoption as standard without revision of the Tentative Method for Sampling Bulk Shipments of Carbon Black (D 1900).

Subcommittee VII on Specification (J. H. Gifford, chairman) is continuing work on methods of tests for carbon blacks used in styrene-butadiene rubbers (SBR). The work is in conjunction with Subcommittee XXIX on Compounding Ingredients of Committee D-11.

This report has been submitted to letter ballot of the committee, which consists of 43 members; 30 members returned their ballots, of whom 28 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. L. HUTSON,
Chairman.

T. D. BOLT,
Secretary.

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

REPORT OF COMMITTEE D-26

ON

HALOGENATED ORGANIC SOLVENTS*

Committee D-26 on Halogenated Organic Solvents held two meetings during the year: in New York, N. Y., on June 28 and 29, 1962, and in Montreal, Quebec, on Feb. 7 and 8, 1963. A number of subcommittees and task groups met in conjunction with the main committee and between meetings of the main committee.

The committee consists of 93 members, of whom 40 are classified as producers, 33 as consumers, and 20 as general interest members. In addition, there are 14 consultants.

The officers elected in 1962 for the ensuing term of two years are as follows:

Chairman, W. D. McMaster.
Vice-Chairman, N. E. Whitman.
Secretary, C. L. Cormany.
Executive Subcommittee Member-at-Large, W. Campbell.

During the year, the committee lost because of death the services of H. Wochele, a charter member.

Tentative Continued Without Change

The committee recommends the continuation without revision of the Tentative Method of Test for Relative Evaporation Time of Halogenated Hydrocarbon Solvents and Their Admixtures (D 1901 – 61 T).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Vapor Degreasing (V. B. Smesrude, chairman) sponsored a Symposium on Vapor Degreasing at the Pacific Area National Meeting in Los Angeles, Calif. in October, 1962.

Task groups have been working on the formulation of property and performance specifications for perchloroethylene and trichloroethylene suitable for vapor degreasing service, and assisting governmental agencies in the revision of existing Federal Specifications.

Subcommittee III on Cold Cleaning (M. Poliakoff, chairman) has nearly completed work on a manual covering all phases of cold cleaning.

A task group has been created to study the use of cold cleaning solvents in the electrical industry and the possible need for specifications covering specialty cleaning formulations.

Subcommittee IV on Test Methods (E. G. Richardson, chairman).—Task groups have pursued the development of test methods requested by other subcommittees. Six such methods have been issued by the Society; three are under consideration, and are nearly ready for ballot. Existing ASTM methods were first evaluated by the committee to determine suitability for use and reference.

Subcommittee V on Industrial Hygiene

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

(N. E. Whitman, chairman) has been assisting Subcommittee III in the preparation of the cold cleaning manual.

Subcommittee VI on Textile Drycleaning (L. C. Kienzle, chairman).—The organization of this subcommittee has been completed and a scope of interest determined. Task groups have been appointed to formulate drycleaning solvent specifications, investigate safety considerations, study current zoning regulations, and prepare a history of drycleaning.

Respectfully submitted on behalf of the committee,

WARDLEY D. MCMASTER,
Chairman.

C. L. CORMANY,
Secretary.

REPORT OF COMMITTEE D-27
ON
ELECTRICAL INSULATING LIQUIDS AND GASES*

Committee D-27 on Electrical Insulating Liquids and Gases held three meetings during the year: in New York, N. Y., on June 27–29, 1962, during the Annual Meeting; in Pittsburgh, Pa. on Oct. 7–10, 1962; and in Memphis, Tenn. on March 17–20, 1963.

The committee consists of 106 members, of whom 97 are active, and 73 are voting representatives. The composition of the membership is: 29 producers, 51 consumers, 16 General Interest members, 9 Inactive members, and 1 Honorary member.

Liaison representation on other ASTM committees and other organizations by members of Committee D-27 continues to be important in coordinating projects.

J. C. Parker has resigned as the representative of Committee D-27 on Committee D-9 on Electrical Insulating Materials. His successor is F. P. West.

George Shombert, Jr., reports regularly to Committee D-27 on the activities of Committee E-11 on Quality Control of Materials.

John G. Miller continues to bring news of the activities of Committee D-2 on Petroleum Products and Lubricants as they pertain to Committee D-27 methods and subjects.

Earl R. Thomas, the American Technical Advisor on Insulating Oils to the U. S. Committee of the International Electrotechnical Commission (IEC), and an American representative on the Committee on Large Electrical Systems [CIGRE], went abroad in May, 1962, to attend the meeting of Study Committee No. 1 on Insulating Oils [CIGRE] in Paris. Mr. Thomas recommended to the U. S. National Committee of IEC that both the IEC Method for Oxidation Testing of Insulating Oils as well as the IEC Dielectric Test Procedure be accepted for use on oils for international trade. A start was made on an attempt to prepare an international specification for insulating oil of petroleum origin by assembling a large number of specification lists used by governments, manufacturers of apparatus, refiners, and utility operators. These data are now being studied.

Mr. Thomas also proposed that some future annual meeting of Technical Committee 10 of the IEC be held in the United States simultaneously with a meeting of Committee D-27 so that there could be a mutual exchange of ideas on study projects and technical organization. This is several years away at the present, but is being given consideration.

The scope of Technical Committee 10 of the IEC has been under discussion for some time as there is a limitation to prepare only a method of test for insulating oils to evaluate their expected performance in service. It had been proposed by Mr. Thomas that the scope be modeled after that of Committee D-27 to include the study of liquids and gases and to prepare appropriate test methods to evaluate their qualities.

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* Sixty-sixth Annual Meeting of the Society June 23–28, 1963.

With the recommendation that the IEC Method for Oxidation Testing be accepted for international trade, a task group in Committee D-27 is attempting to include the salient features of ASTM Method D 1314 (sludge by accumulation) and of the IEC oxidation test in one new method which might be acceptable to both IEC and ASTM.

In the case of the IEC Method for Dielectric Strength, it is believed that the permissible stirring of the oil during the application of voltage is a sounder technical approach to the determination of the actual condition of the oil.

Although a general meeting of the IEC has been proposed for May, 1963, in Venice, it will not be practicable to hold a meeting of Technical Committee 10 until the scope statement has been made acceptable.

On Oct. 8, 1962, Committee D-27 held a Symposium on Electrical Insulating Gases in Pittsburgh, Pa. under the direction of Vernon R. Mulhall. Four papers were presented with introductory remarks by M. L. Manning, Dean of Engineering of the South Dakota State College. These papers will be published by the Society as a Special Technical Publication.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1962 Annual Meeting, Committee D-27 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Method of Test for:

Color of Chlorinated Aromatic Hydrocarbons (Askarels) (D 2129 – 62 T),
Examination of Electrical Insulating Oils by Infrared Absorption (D 2144 – 63 T), and
Carbon-Type Composition of Insulating Oils of Petroleum Origin (D 2140 – 63 T).

Revision of Tentative Method for:

Visual Examination of Used Electrical Insulating Oils in the Field (D 1524 – 58 T),
Sampling Askarels (D 1809 – 60 T), and
Visual Examination of Used Askarels in the Field (D 1702 – 59 T).

Revision and Reversion to Tentative of Standard Methods of Test for:

Power Factor and Dielectric Constant (D 924 – 58), and
Corrosive Sulfur in Electrical Insulating Oils (D 1275 – 58).

The new Tentative Method D 2129 was accepted by the Standards Committee on October 24, 1962, and is available as a separate reprint. The new Tentative Methods D 2144 and D 2140, and revised methods D 924 and D 1524 were accepted on March 24, 1963. The revised methods D 1275, D 1702, and D 1809 were accepted on April 29, 1963.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee D-27 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] All of the existing tentatives and standards referred to appear in their present form in the 1961 Book of ASTM Standards, Part 11, or the 1962 Supplement thereto.

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[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Adoption of Tentatives as Standard Without Revision

Tentative Methods of Test for:

Scavenger Content of Askarels (**D 1701 - 59 T**),
Volume of Oil in Oil-Contaminated Askarels (**D 1808 - 60 T**),
Specific Gravity of Askarels (**D 1810 - 60 T**),
Inorganic Chlorides in Askarels (**D 1821 - 60 T**), and
Water in Insulating Oils by Extraction (**D 1315 - 59 T**).

Tentative Recommended Practice for:

Estimating Relative Temperature Limits of Dielectric Liquids (**D 1805 - 60 T**).

Tentative Continuity Specifications for:

Electrical Insulating Oil for Low-Pressure Cable Systems (**D 1818 - 60 T**), and
Electrical Insulating Oil for High-Pressure Cable Systems (**D 1819 - 60 T**).

Adoption of Tentative Revision as Standard

Standard Methods of Testing Askarels (**D 901 - 62**):

New Sections 53 to 55.—Incorporate as standard new Sections 53 to 55 on water content, issued Sept. 15, 1959.

Tentatives Continued Without Revision

The committee recommends that those tentatives that have stood for two years or more without revision, which are not mentioned earlier in the report, be continued in their present form pending future revision.

ACTIVITIES OF SUBCOMMITTEES

Materials Subcommittee A on Mineral Oils (E. L. Raab, chairman) contains three main groups. The first group is surveying the general problems of transformer operation. Progress is being made in examining the several methods for detecting incipient faults in oil-filled transformers by analyzing the gas cushion above the oil. A suitable method for the detection of combustible gases in this gas mixture must be devised, which involves examining the various explosion meters now available. Methods of sampling the gas cushion will be required. The effect of CO_2 on the oil in service is being investigated as an increase in pressure within a transformer greatly increases gas solubility in the oil. It is hoped that the analysis of the gas found may lead to its source.

The second group on transformer oils has undertaken a comprehensive revision of the Recommended Practice for the Purchase of Insulating Oil (D 1040) to see if this cannot be made more severe in its requirements without illegally restricting marginal suppliers. It is being considered whether one set of specification limits would be sufficient or if several types of oils must be covered by different sets of test standards. Most important is the inclusion of an aging test which may be correlated with the service life of an oil, a goal which still seems far removed from the several oxidation tests available. Considerable emphasis is being placed on power factor of oil in service, and methods are being studied to determine if the oils in laboratory tests increase in power factor at comparative rates with oils in service. In interlaboratory studies, power factor increase is becoming a fixed criterion of quality.

The third group is studying oils and other liquid insulating materials for use in cables, capacitors, and accessory appliances. An intermediate viscosity oil specification is being prepared, and vegetable oils, waxes, and other such products used in the liquid state will be included. The two purchase specifications for oils for low-pressure and high-pressure oil-filled cables have been adopted as standard, but no attempt will be made to raise the minimum flash point of the low-pressure type oil.

Materials Subcommittee B on Synthetics (T. K. Sloat, chairman) has prepared a new method for determining the color of askarels, as the oil-color standards had been found inadequate in shade and were too dark to show differences in low colored materials. It has completed also a revision of the Method for Sampling Askarels (D 1809). A specification for askarels for transformer and capacitor use is being prepared with the help of the main supplier of this material. It was felt that the test characteristics would be most valuable to purchasers.

The group studying silicones has found that many of the oil test methods can be applied to silicones without change, and others will be acceptable with only slight changes or additions to the procedures. Where new test methods must be devised, this work will be referred to the appropriate method subcommittee. It is an aim of this group to compile a set of methods for silicones, similar to D 117 on Transformer Oils and D 901 on Askarels.

Materials Subcommittee C on Gases (George Shombert, Jr., chairman) is preparing a specification for sulfur hexafluoride. With the completion of the work on nitrogen (Specification D 1933), the preparation of a similar type specification for hydrogen as a cooling and insulating material is being considered and if the demand is sufficient, a specification will be prepared. Methods for sampling these gases will be prepared by Subcommittees O and P. A survey is under way to determine if there is sufficient use as yet of the fluorocarbon gases to warrant work on test methods and specifications. In this new field of materials, there are few users familiar with the products and their characteristics, and the producers must be represented on the committee for the development of the required test procedures.

Methods Subcommittee N on Electrical Tests (H. O. Bennett, chairman) has five active sections. A review of the method for power factor and dielectric constant has been under way for more than two years and is nearing completion. The section on dielectric strength has again run into difficulty and the rewriting of Method D 877 has become a much larger project than originally considered. There is still no widespread acceptance of Method D 1816 using the VDE gap and electrodes as a routine procedure.

The section on gas evolution has been hampered by several necessary changes in its leadership, but it is now studying gas cells available and other pieces of apparatus not encumbered by patent restrictions.

The section studying the stability of liquid impregnated paper has found it advantageous to work closely with Subcommittee VIII on Electrical Paper of Committee D-9 on Electrical Insulating Materials which is interested in the change in the condition and quality of the paper in the oil-paper combination, whereas the deterioration of the oil is the objective of our operators. Interlaboratory tests are being run by both groups, and a mass of data is being correlated.

The dielectric strength of gases required new methods of measurement different from the procedures on liquids. Several cells have been devised and circulated for trial. A paper was presented at the symposium in October, 1962, on this subject and was received with interest.

Some work has also been started on determining the dielectric strength of synthetic fluids where the amounts required must be restricted because of their extreme cost. This involves the development of micro-apparatus, of which the cleaning and operation are being carefully studied.

Methods Subcommittee O on Chemical Tests (H. W. McCulloch, Jr., chairman) has completed a revision of the Method of Test for Corrosive Sulfur in Electrical Insulating Oils (D 1275) and believes that the new terminology will make it possible for different operators to agree closely in their evaluation of corrosion effects by this method.

The preparation of an oxidation procedure which will embody the salient features of the modified turbine oil oxidation test method, the Sludge by Accumulation Test (D 1314), and the Test for Aging Characteristics (D 1904), is still under way and has proved to be a formidable task. Work is also continuing on a study to combine the IEC aging test and Method D 1314, for sludge by accumulation.

A new section has been authorized to prepare a test method for determining the hydrolizable fluorides in sulfur hexafluoride which is an indication of the widening range of procedures required as the study of insulating gases expands.

Methods Subcommittee P on Physical Tests (J. C. Parker, chairman) has completed new methods for steam emulsion, for color of askarels, for carbon-type composition of insulating oils, and for the examination of insulating oils by infrared absorption. Three methods have been revised: on the visual examination of used oils, and of used askarels, and the method for refractive index and specific optical dispersion of oils. Sections working on the preparation of methods for determining molecular weight of oils, and for measuring the hydrogen and oxygen content of nitrogen gas report steady progress.

The section investigating volatility of oils found that the flash point of an oil seemed to give sufficient safeguard against the inclusion of light ends in oils which might cause them to lose large amounts by evaporation. The work will be deferred for the present.

New sections on gas chromatography and on the amount of combustible gases and nitrogen (in the gas cushion) over oil have been formed.

Testing methods for gases, synthetics, and new types of insulating fluids will require that the personnel of Subcommittee P, be augmented by additional personnel with special knowledge of these new fields.

This report has been submitted to letter ballot of the committee, which consists of 73 voting members; 60 members returned their ballots, of whom 57 have voted affirmatively, and 0 negatively.

Respectfully submitted on behalf of the committee,

Earl R. Thomas,
Chairman.

Charles A. Johnson,
Recording Secretary.

REPORT OF COMMITTEE D-28
ON
ACTIVATED (ACTIVE) CARBON*

Committee D-28 on Activated (Active) Carbon was organized in Philadelphia, Pa., on Nov. 15, 1962.

The scope of this committee is: "The development of definitions of terms and nomenclature and methods of test for activated (active) carbon and the promotion of research in this field."

The officers elected for the ensuing term of two years are as follows:

Chairman, Earl T. Ellis.
Vice-Chairman, Henry F. Laughlin.
Secretary, G. H. Scheffler.

Three subcommittees were formed as follows:

Subcommittee I on Properties (I. P. Mail, chairman) is collecting existing methods for density, ash, surface area, particle size distribution, and decolorizing properties.

Subcommittee II on Evaluation Tests (F. R. Schwartz, chairman) is drafting general procedures for the liquid and gas-phase adsorption isotherm.

Subcommittee III on Nomenclature and Editorial (J. W. Hassler, chairman) is circulating a list of terms of particular interest to the activated (active) carbon industry in order to develop satisfactory definitions.

Respectfully submitted on behalf of the committee,

Earl T. Ellis,
Chairman.

G. H. Scheffler,
Secretary.

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

REPORT OF ALCA-ASTM JOINT COMMITTEE
ON
LEATHER*

The ALCA-ASTM Committee on Leather (joint with the American Leather Chemists' Assn.) held two meetings during the year: on Oct. 23 and 24, 1962, in Chicago, Ill., in conjunction with a meeting of the Tanners' Council of America, and on March 14 and 15, 1963, in Philadelphia, Pa., in conjunction with a meeting of the Delaware Valley Tanners' Club.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

The ALCA-ASTM Joint Committee on Leather requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] All of the existing tentatives and standards referred to appear in their present form in the 1961 Book of ASTM Standards, Part 6, and the 1962 Supplement thereto.

New Tentatives

as appended hereto[2]

Proposed Tentative Methods of Test for:

Tensile Strength of Leather,
Grain Crack and Extension of Leather by the Mullen Test,
Elongation of Leather,
Breaking Strength of Leather by the Grab Method,
Bursting Strength of Leather by the Ball Method, and
Slit Tear Resistance of Leather.

Adoption of Tentative as Standard Without Revision

Tentative Method of Test for Resistance to Wetting of Garment-Type Leathers (Spray Test) (**D 1913 – 61 T**).

Tentative Continued Without Revision

Tentative Method of Test for Cold-Crack Resistance of Upholstery Leather (**D 1912 – 61 T**).

Editorial Changes

Tentative Method of Test for Colorfastness and Transfer of Color in the Washing of Leather (**D 2096 – 62 T**):

Footnote 6.—Change "Junior" to read "*Journal*" in the list of references.

American Standards

The committee recommends that the following standards and tentatives be submitted to the American Standards Assn. for adoption as American Standard:

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[2] The new tentatives appear in the 1963 Supplement to *Book of ASTM Standards*, Part 6.

Standard Methods of:

Test for Area of Leather (**D 1515 - 60**),
Test for Width of Leather (**D 1516 - 60**),
Conditioning Leather and Leather Products for Testing (**D 1610 - 60**),
Test for Corrosion Produced by Leather in Contact with Metal (**D 1611 - 60**), and
Test for Water Absorption (Static) of Leather (**D 1815 - 62**).

Standard Definitions of:

Terms Relating to Leather (**D 1517 - 60**).

Tentative Methods of Test for:

Bond Strength of Leather Belting (**D 1699 - 60 T**),
Measuring Thickness of Leather Test Specimens (**D 1813 - 62 T**),
Measuring Thickness of Leather Units (**D 1814 - 62 T**),
Cold-Crack Resistance of Upholstery Leather (**D 1912 - 61 T**),
Resistance to Wetting of Garment-Type Leathers (Spray Test) (**D 1913 - 61 T**),
Colorfastness and Transfer of Color in the Washing of Leather (**D 2096 - 62 T**),
Dynamic Water Resistance of Shoe Upper Leather by the Dow Corning Leather Tester (**D 2098 - 62 T**), and
Dynamic Water Resistance of Shoe Upper Leather by the Maeser Water Penetration Tester (**D 2099 - 62 T**).

Tentative Method of:

Flex Testing of Finish on Upholstery Leather (**D 2097 - 62 T**).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature, Definitions, and Applicability (R. M. Lollar, chairman) has (1) approved the drafts of the six proposed tentatives developed by Subcommittee A3 on Tensile Properties; (2) approved the draft of the Tentative Method for Determination of Thermal Conductivity Constant of Leather with the Cenco-Fitch Apparatus and the draft of the Method of Test for Compressibility of Leather as developed by Subcommittee A10 on Miscellaneous Physical Properties; and (3) returned to Subcommittee A8 on Deterioration the revised draft of the Method for Drycleaning Test for Suede Leather.

Subcommittee II on Sampling and Conditioning for Physical Testing was inactive due to resignation of the chairman. It is planned to reactivate this group when a new chairman is appointed.

A. General Physical Testing Group:

Subcommittee A2 on Physical Dimensions (L. Seligsberger, chairman) has developed a draft of a procedure for measuring the apparent density of leather and is studying procedures for measuring (1) areas of leather specimens for use when it is desired to determine changes in area resulting from exposure to test environments; (2) the real and apparent densities of leathers in order to determine porosity; and (3) the stiffness of leather.

Subcommittee A3 on Tensile Properties (R. L. Young, Jr., chairman) has developed the six new tentative methods for tensile properties of leather as appended to this report.[2]

Subcommittee A4 on Measurement of Water Resistance (M. Brown, chairman) has (1) recommended that Method D 1913 be adopted as standard without revision; (2) continued studies on the reliability of results using dynamic water testers; (3) tabled further study on the comparison of tap tester with dynamic tester; and (4) decided that further work was needed on the method for water vapor transmission.

Subcommittee A5 on Surface Characteristics (Robert Stubbings, chairman) has (1) recommended that Method D 1912 be continued as tentative without revision; (2) initiated interlaboratory studies on the evaluation of five methods for measuring wet and dry crock resistance of six different leathers; (3) continued work on a method of test for scuff resistance of leather using an attachment to the standard crock meter; and (4) recommended that the bulk data from the lightfastness tests be filed in the ASTM research reports file and that a summary be published.

Subcommittee A8 on Deterioration (P. Beuchler, chairman) has (1) recommended that the Proposed Method of Measuring Resistance of Chrome-Tanned Shoe Upper Leather to Artificial Perspiration be published as information only[3]; (2) undertaken the development of a universal perspirant solution so that method could be extended to include vegetable-tanned leathers; and (3) taken over the responsibility for mechanical leathers from the disbanded Subcommittee A7.

Subcommittee A10 on Miscellaneous Physical Properties (M. Bailey, chairman) conducted an interlaboratory abrasion test on sole leather. Variance between instruments was found to be excessive, but despite these differences Army and Navy testers showed that wet wear is more severe than dry wear. Equipment is being modified to reduce instrument variances. The draft of method for compressibility will be submitted to letter ballot. Interlaboratory tests are in progress to develop precision data for the method for measuring break.

B. Chemical Testing Group (W. T. Roddy, organizing chairman) established two subcommittees to study and prepare standards in this area. Other subcommittees may be formed as the need arises and personnel become available.

Subcommittee B1 on Mineral Leathers (N. Clifford Benrud, chairman) has been organized and the membership assigned methods for review and consideration of suitability for standards. Exploration of methods for measuring shrinkage temperature was reported.

Subcommittee B2 on Oils and Greases (M. Leach, chairman) was organized and several member groups are actively drafting methods on this area. It was found that of the 53 ALCA methods, 28 are either essentially the same as ASTM methods for similar materials or modifications.

Subcommittee IV on Research (M. M. Baldwin, chairman) reviewed several new areas pertaining to leather properties and characteristics: (1) L. P. Witnauer investigated the effect of temperature on the stiffness of soling materials and reported that leather showed the least variation of the materials studied; (2) further consideration of torsional properties of leather was assigned to Subcommittee A10 on Miscellaneous Properties; (3) means of evaluating the reaction of leather with finishes to heat, moisture, and stretch was assigned to Subcommittee 5 on Surface Characteristics for further study; (4) a study group was formed to explore and define the problems associated with measurement of the dynamic properties of leather required for proper shoe fabrication; (5) the porosity of leather was assigned to Subcommittee A2 on Physical Dimensions for further study; and (6) a study of the comparative slipperiness of sole leather could evolve into a measurement of the coefficient of friction. The question was raised of expanding the scope of the joint committee to include development of test methods for evaluating fabricated parts and Messrs. Young and Bailey were appointed to study the question further. The latest developments of the International Union [of Leather Chemists]

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[3] Withdrawn as a consequence of letter ballot of the Joint Committee.

Physical Testing Commission were reviewed.

Subcommittee V, Editorial (J. R. Kanagy, chairman) has concerned itself principally with the form and organization of test methods.

This report has been submitted to letter ballot of the committee, which consists of 35 members; 27 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. NAGHSKI,
Chairman.

M. BAILEY
Secretary.

REPORT OF COMMITTEE E-1

ON

METHODS OF TESTING*

Committee E-1 on Methods of Testing held a meeting in Philadelphia, Pa. on Dec. 4, 1962. During the June, 1962, Annual Meeting in New York, N. Y., there were meetings of 40 subcommittees and sections. Subcommittee 17 on Thermometers and Hydrometers met in New York, N. Y., on Oct. 26, 1962. Meetings of Task Group B on Linear Thermal Expansion Test were held in Philadelphia, Pa., on May 16, 1962, and March 14, 1963. Meetings were held of ten subcommittees and sections during ASTM Committee Week in Montreal, Canada, from Feb. 4 to 8, 1963.

A Symposium on Resinographic Methods is being sponsored by Subcommittee 28 on Microscopy (T. G. Rochow, chairman) at the 1963 Annual Meeting; the 18 papers in this symposium will be presented in four technical sessions on June 27 and 28.

The work on thermometers under Subcommittee 17 and the activities of Subcommittee 32 on Thermocouples for Temperature Measurement were transferred to the new ASTM Committee E-20 on Temperature Measurement.

The following three new subcommittee chairmen were appointed during the year: F. A. Lobisser, The George Ruehfel Co., Inc., chairman of Subcommittee 18 on Hydrometers; E. H. Enberg, Riehle Testing Machine Div., Ametek, Inc., chairman of Subcommittee 30 on Bend Testing; and Prof. A. H. Corwin, Johns Hopkins Univ., chairman of Subcommittee 31 on Laboratory Balances.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1962 Annual Meeting, Committee E-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specifications for:

Oxygen Combustion Flask (E 205 – 62 T)

New Tentative Method of:

Thermal EMF Test of Single Thermoelement Materials by Comparison with a Secondary Standard of Similar EMF-Temperature Properties (E 207 – 62 T).

Revision of Tentative Definitions of Terms Relating to:

Microscopy (E 175 – 61 T).

Tentative Revision of Standard Definitions of Terms Relating to:

Methods of Mechanical Testing (E 6 – 62).

The new tentatives E 205 and E 207 and the tentative revision of Definitions E 6 were accepted on Oct. 17, 31, and 19,

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

1962, respectively, and they appear in the 1962 Supplement to Book of ASTM Standards. The revision of Tentative Definitions E 175 was accepted on March 21, 1963, and has been issued as a separate reprint.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee E-1 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] All of the existing tentatives and standards referred to appear in their present form in the 1961 Book of ASTM Standards, or the 1962 Supplement thereto.

New Standard

Standard Microscope Objective Thread, appended hereto.[2]

This standard was developed by ASA Sectional Committee B1 on Standardization and Unification of Screw Threads, under the sponsorship of The American Society of Mechanical Engineers and the Society of Automotive Engineers. Permission has been obtained from the ASME and SAE to publish this as an ASTM Standard. This microscope objective thread is a basic part of a microscope and will serve as a foundation for other standards to be prepared by Section 2 on Microscopes and Their Elements (O. W. Richards, chairman) of Subcommittee 28 on Microscopy.

New Tentative

Tentative Specifications and Methods of Test for Cover Slips and Glass Slides for Use in Microscopy, as appended hereto.[2]

Revision of Standard, Immediate Adoption

(requires nine-tenths affirmative vote)

Standard Definitions of Terms Relating to Methods of Mechanical Testing (**E 6 – 62**):

Add the following two new definitions under Section B:

Modulus of Rupture in Bending.—The value of maximum tensile or compressive stress (whichever causes failure) in the extreme fiber of a beam loaded to failure in bending computed from the flexure formula:

$$S_b = \frac{Mc}{I}$$

where:

M = maximum bending moment, computed from the maximum load and the original moment arm,

c = initial distance from the neutral axis to the extreme fiber where failure occurs, and

I = initial moment of inertia of the cross-section about the neutral axis.

NOTE 1.—When the proportional limit in either tension or compression is exceeded, the modulus of rupture in bending is greater than the actual maximum tensile or compressive stress in the extreme fiber, exclusive of the effect of stress concentration near points of load application.

NOTE 2.—If the criterion for failure is other than rupture or attaining the first maximum of load, it should be so stated.

Modulus of Rupture in Torsion.—The value of maximum shear stress in the extreme fiber of a member of circular cross-section loaded to failure in torsion computed from the formula:

$$S_s = \frac{Tr}{J}$$

where:

T = maximum twisting moment,

r = original outer radius, and

J = polar moment of inertia of the original cross-section.

NOTE 1.—When the proportional limit in shear is exceeded, the modulus of rupture in torsion is greater than the actual maximum shear stress in the extreme fiber, exclusive of the effect of stress concentration near points of application of torque.

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[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[2] The new standard and the new tentative appear in the 1963 Supplement to *Book of ASTM Standards*, Part 3.

NOTE 2.—If the criterion for failure is other than fracture or attaining the first maximum of twisting moment, it should be so stated.

Adoption of Tentative Revision as Standard, With Revisions

Standard Definitions of Terms Relating to Methods of Mechanical Testing (E 6 - 62):

Adopt as standard the five definitions issued as tentative revisions Oct. 19, 1962, revised as follows:

14A. **Set.**—Strain remaining after complete release of the load producing the deformation.

NOTE 1.—Due to practical considerations such as distortion in the specimen and slack in the strain indicating system, measurements of strain at a small load rather than zero load are often taken.

NOTE 2.—Set is often referred to as permanent set if it shows no further change with time. Time elapsing between removal of load and final reading of set should be stated.

20. **Breaking Load.**—The load at which fracture occurs (which may be less than the maximum load).

NOTE.—When used in connection with tension tests of thin materials or materials of small diameter for which it is often difficult to distinguish between the breaking load and the maximum load developed, the latter is considered to be the breaking load.

23. **Fatigue.**—The process of progressive localized permanent structural change occurring in a material subjected to conditions which produce fluctuating stresses and strains at some point or points and which may culminate in cracks or complete fracture after a sufficient number of fluctuations.

NOTE 1.—The term fatigue in the materials testing field, has, in at least one case, glass technology, been used for static tests of considrable duration, a type of test generally designated as stress-rupture.

NOTE 2.—Fluctuations may occur both in stress and with time (frequency) as in the case of "random vibration."

32. **Stress Ratio,** A or R.—The algebraic ratio of two specified stress values in a stress cycle. Two commonly used stress ratios are:

The ratio of the stress amplitude to the mean stress, that is,

$$A = \frac{S_a}{S_m}$$

and the ratio of the minimum stress to the maximum stress, that is,

$$R = \frac{S_{\min}}{S_{\max}}$$

57. **Creep.**—The time-dependent part of strain resulting from stress.

NOTE 1.—Instantaneous strains are excluded.

NOTE 2.—Creep is usually measured under constant load, but measurements may be made under conditions of constant stress or under special conditions where both load and stress may vary. Where the total strain is small, the difference between constant load and constant stress tests is negligible, but for larger strains, corrections must be made in results of constant load tests, or the fact that the value is reported for constant load tests must be noted.

(*a*) *Primary Creep:*

(*1*) The strain occurring at a diminishing rate.

NOTE.—This term has also been used to denote the recoverable portion of creep.

(*2*) Delete.

(*b*) *Secondary Creep:*

(*1*) The strain occurring at a constant rate.

NOTE.—This term has also been used to denote the non-recoverable portion of creep commonly called permanent set.

(*2*) Delete.

(*c*) *Tertiary Creep.*—The strain occurring at an accelerating rate.

Adoption of Tentative Revision as Standard, Without Revision

Definitions of Terms Relating to Methods of Mechanical Testing (E 6 - 62):

Section 34.—Adopt definition of the term "Stress Cycles Endured."

Adoption of Tentatives as Standard Without Revision

Tentative Definitions of Terms Relating to Conditioning (**E 41 - 60 T**),

Tentative Specifications for Standard Atmospheres for Conditioning and Testing Materials (**E 171 - 60 T**)

Revision of Tentatives

Tentative Methods of Test for Water Vapor Transmission of Materials in Sheet Form (E 96 - 53 T):

Revise as appended hereto.[3]

Tentative Recommended Practice for Operating Enclosed Carbon-Arc Type Apparatus for Artificial Light Exposure Tests (E 188 - 61 T):

Section 3.—Under Paragraph (*a*) (*1*) change Footnote 4 to read as follows:

[4] Available from Atlas Electric Devices Co., 4114 N. Ravenswood Ave., Chicago 13, Ill. The Model FDA-R Fade-Ometer with intermittent blower operation but not equipped with an atomizer unit meets the requirement of Method A only. The Model FDA-RC Fade-Ometer with intermittent blower operation and an atomizer unit and the Models 18-F, 18-FT and 18-FR Fade-Ometer or equivalent with continuous blower operation and manual or automatic control of humidity meets the requirements of either Method A or Method B.

In Paragraph (*a*) (*2*) change the last sentence to read, "The framework with specimens shall revolve about the arc 2 ± 1 times a minute."

Delete Paragraph (*a*) (*3*) and renumber the present (*a*) (*4*) as (*a*) (*3*).

Add a new Paragraph (*a*) (*4*) to read as follows:

3. (*a*) (*4*) A blower unit in the base of the apparatus provides a flow of air over the test specimens and through the test chamber. Control of specimen and black panel temperatures is preferably accomplished by continuous operation of the blower with thermostatic control of the temperature of the air, but the use of room temperature air whose volume is thermostatically regulated by on-off operation of the blower is permissible. Where temperatures are controlled by regulating the temperature of a constant volume of air, an electrically operated atomizing unit with a capacity of one gallon per hour adds moisture to the air as it passes through the conditioning chamber in the base section of the apparatus prior to its entry into the test chamber. Unless otherwise mutually agreed upon, the relative humidity in this type of apparatus is adjusted to 30 ± 5 per cent as measured by wet and dry bulb thermometers at the point of exit of air from the test chamber. The type of apparatus providing control of temperature by intermittent operation of the blower if also equipped with an electrically operated atomizing unit shall for the purposes of this method, unless otherwise mutually agreed upon, be operated with the atomizing unit shut off. The relative humidity of the air in the test chamber thus is not controlled but is that which is produced by the use of room air which is cooled, filtered, and humidified by passing over wicks or sponges saturated with water.

Note 2.—In the first sentence change "seven" to "eight" and "L-8" to "L-9."

Section 4.—In Paragraph (*a*) change the first sentence to read: "Check to be sure that the apparatus is in good running order at the start of each test or 24-hr period and is equipped with a clean globe free of chips and cracks and a new trim or set of carbons."

In Paragraph (*c*) delete the last sentence as this is now covered by the revised Section 3 (*a*) (*4*) given above.

Note 4.—In the twentieth line delete the words "above the level of 0.823 g cal per sq cm."

Section 7(a).—Change to read as follows:

(*a*) The apparatus if employing intermittent operation of the blower shall be additionally equipped with an electrically operated atomizing unit with a capacity of 2 pt of water per hour for increasing the moisture content of the air in the test chamber. Unless otherwise mutually agreed upon, the atomizer shall be in operation continuously during both the light-on and light-off periods. The relative humidity of the air passing through the test chamber is not automatically controlled but is that produced by room air passing over wicks saturated with water to which moisture is added by the atomizing unit. The apparatus if employing continuous operation of the blower is also equipped with an electrically operated atomizing unit with a capacity of one gallon per hour for adding moisture to the air as it passes through the conditioning chamber in the base section of the apparatus prior to its entry into the test chamber. Unless otherwise mutually agreed upon, the relative humidity after stabilization is reached shall be manually adjusted or automatically controlled at 35 ± 5 per cent during the light-on period and 90 ± 5 per cent during the light-off period as measured by wet- and dry-bulb thermometers at the point of exit of air from the test chamber.

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[3] The revised tentative appears in the 1963 Supplement to *Book of ASTM Standards*, Parts 4, 5, 6, 10.

Section 8.—In the second paragraph, third sentence, change "(Note 2)" to read "(Note 3)," add a period, and delete the remainder of the sentence.

Editorial Changes

Standard Specifications for ASTM Hydrometers (**E 100 – 62**):

Note 1.—Change the second sentence to read: "Hydrometers manufactured prior to the adoption of the specifications will retain the same official status as those meeting current specifications."

Table I.—Under Hydrometer 152H-62 change the Standard Temperature from "68 F" to "68/68 F."

American Standards

The committee recommends that the following standards be submitted to the American Standards Assn. for approval as American Standards:

Definitions of Terms Relating to Conditioning (**E 41 – 63**),

Specifications for Standard Atmospheres for Conditioning and Testing Materials (**E 171 – 63**), and

Method for Determining Relative Humidity by Wet- and Dry-Bulb Psychrometer (**E 337 – 62**).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee 1 on Calibration of Mechanical Testing Machines and Apparatus (B. L. Wilson, chairman).—Revisions in the Method of Verification and Classification of Extensometers (E 83) are in preparation by Section 3 on Calibration and Verification of Extensometers (R. H. Heyer, chairman). The Section on Evaluation of Remote Reading Strain Gages (R. L. Bloss, chairman) has in preparation drafts of proposed methods for determining the following characteristics: (1) gage factor, (2) variation of gage factor with temperature, (3) transverse sensitivity, (4) temperature sensitivity, and (5) leakage resistance.

Subcommittee 2 on Effect of Speed in Mechanical Testing (L. K. Irwin, chairman).—A Method for Determining Errors in Tension Testing Machines Due to Speed of Testing has been prepared and is being circulated to the membership with a letter ballot for publication as tentative.

Subcommittee 3 on Elastic Properties and Definitions on Mechanical Testing (G. R. Gohn, chairman).—Section 1 on Elastic Constants (J. A. Miller, chairman) and the subcommittee have reviewed the latest draft of the Tentative Method for the Static Determination of Young's Modulus at Low and Elevated Temperatures which has been prepared by the Joint ASME-ASTM Committee on Effect of Temperature on the Properties of Metals. This review has indicated desirable minor changes in the present draft before publication by the Society as tentative.

Section 2 on Review of Definitions (James Quigley, chairman) has recommended that the tentative definitions for fatigue, stress ratio, stress cycles endured, breaking load, and set in Definitions E 6 be adopted as standard. Editorial changes in the first three definitions are recommended so that the wording will agree with that given in the definitions as now published in Definitions E 206. It is further recommended that the definition for creep be revised to reflect changes made in the section dealing with primary, secondary, and tertiary creep and that the revised definition be adopted as standard. Two new definitions on modulus of rupture in bending and on modulus of rupture in torsion are also recommended for adoption and inclusion in Definitions E 6. Under consideration are definitions for uniaxial, biaxial, and triaxial stress, true strain, true stress, true stress-strain, diagram, ductility, fiber stress, fracture stress, shear fracture, torsion, and uniform stress, as well as additional abbreviations and symbols for inclusion in the Appendix to Definitions E 6. Special attention is also being given to the inclusion of definitions on mechanical testing practices which appear in various specifications under the jurisdiction of the "C" and "D" committees of the Society.

The subcommittee has reviewed and commented on the proposed Standard Load Cell Terminology and Definitions prepared by the Scientific Apparatus Makers Assn. Draft ISO Document No. 514, "List of Recommended Symbols to be Used in Symbolizing Mechanical Values" has also been reviewed. It is hoped that the latter can eventually be reconciled with the symbols now appearing in Definitions E 6.

Subcommittee 4 on Tension Testing (F. M. Howell, chairman).—Progress is being made on a revision of the Methods of Tension Testing of Metallic Materials (E 8) to provide methods mutually acceptable to the various ASTM metals committees and to those responsible for Federal Test Method 211. This work is being carried on by Section 1, which is now under the chairmanship of J. M. Quigley.

Section 2, under the chairmanship of C. H. Marshall, has under consideration requirements for one or more pin-loaded tension specimens.

Subcommittee 5 on Compression Testing (J. A. Miller, chairman).—A proposed Method of Test for Compressive Strength of Lightweight Insulating Concrete, prepared by Committee C-9 on Concrete and Concrete Aggregates, was reviewed and comments were forwarded to Committee C-9.

A proposed Recommended Practice for Compression Tests of Metallic Materials at Elevated Temperatures with Conventional or Rapid Heating Rates and Strain Rates, prepared by a task group of the Test Methods Panel of the Joint ASME-ASTM Committee on Effect of Temperature on the Properties of Metals, was reviewed. Numerous comments and suggestions were submitted to the task group for consideration.

A section is being formed with A. A. Moore as chairman for preparing a method for the determination of bearing strength.

Subcommittee 6 on Indentation Hardness (R. H. Heyer, chairman).—The Section on Hardness-Tensile Strength Conversions for Steel (W. H. Mayo, chairman) is restudying conversions in the hardness range from 20 to 40 Rockwell C scale. Recommendations for changes in the conversion tables of Methods A 370 have not, as yet, been made.

The subcommittee has reviewed several ISO documents on hardness testing.

Subcommittee 7 on Impact Testing (W. W. Werring, chairman).—An extensive revision of the Methods for Notched Bar Impact Testing of Metallic Materials (E 23) has been completed by the Section on Calibration of Impact Machines (N. H. Fahey, chairman). This revision represents a considerable rearrangement with the addition of new material and notably the introduction of the use of standard specimens for the calibration of Charpy impact machines. These standard specimens have been developed by and will be available from Watertown Arsenal.

Subcommittee 8 on Flexural Strength Testing (C. E. Work, chairman) has set up four sections to enable it to review all ASTM tentatives and standards which involve flexure testing in the hope that conflicting nomenclature can be minimized and recommendations can be made to improve and further standardize methods of testing. The subcommittee continues to act in a consulting capacity on flexural strength aspects of standards being developed by other committees.

Subcommittee 9 on Rheological Properties (W. F. Fair, Jr., chairman).—The Section on Softening Point under its new chairman, F. A. Brill, is awaiting the completion of studies under way in Committee D-4 on Road and Paving Materials of the Tentative Method of Test for Softening Point of Asphalts and Tar Pitches (Ring-and-Ball Apparatus) (D 36 - 62 T). Consideration will then be given to appropriate changes in the Method of Test for Softening Point by Ring-and-Ball Apparatus (E 28).

The Section on Brookfield Viscosity under its new chairman, Christian Johnston, is giving consideration to a recommended practice for measuring apparent viscosity by a rotating spindle apparatus (Brookfield viscometer) of Newtonian materials. It is hoped that this can later be expanded to cover non-Newtonian materials.

Subcommittee 11 on Sub-Sieve Testing (L. T. Work, chairman) is preparing a centrifugal sedimentation method for sub-sieve particle size measurement.

Subcommittee 12 on Methods for Density (R. B. Parker, Jr., chairman) is working with ASTM technical committees in seeking unification of terminology, notation, and procedure in the various methods for density determination.

Editorial changes in the Definitions of Terms Relating to Density and Specific Gravity of Solids, Liquids, and Gases (E 12) have been prepared prior to recommending adoption as standard.

The subcommittee has reviewed the Tentative Method of Test for Bulk Density and Porosity of Granular Refractory Materials by Mercury Displacement (C 493) prepared by Committee C-8, the Tentative Methods of Test for Specific Gravity of Halogenated Organic Solvents and their Admixtures (D 2111) prepared by Committee D-26, and the Proposed Method of Test for Density of Loose Fill Insulations prepared by Committee C-16.

Subcommittee 14 on Conditioning and Weathering (F. M. Gavan, chairman).—This subcommittee prepared the revised methods for water vapor transmission (E 96) appended to this report.[3]

The revision of the Tentative Recommended Practice for Operating Enclosed Carbon-Arc Type Apparatus for Artificial Light Exposure Tests (E 188) is proposed to provide for use of the newer types of Fade-Ometers.

It is believed that the proposed revision under study in the Specifications for Standard Atmospheres for Conditioning and Testing Materials (E 171) can be effectively expanded into a new method of specifying the performance characteristics of controlled temperature and humidity cabinets, enclosures, and rooms. Further work will be done by the task group on developing such a method.

Subcommittee 16 on Thickness Measurement (S. B. Newman, chairman).—Review of the first draft of a proposed revision to broaden the scope of the Tentative Methods of Test for Thickness of Solid Electrical Insulation (D 374) resulted in a number of objections and suggested changes. The objections were largely based on omissions of certain test procedures. A new draft is being prepared for subcommittee consideration.

Subcommittee 17 on Thermometers and Hydrometers (J. F. Swindells, chairman).—The activities of this subcommittee on thermometers have been transferred to the new ASTM Committee E-20 on Temperature Measurement, including jurisdiction for the Standard Specifications for ASTM Thermometers (E 1) and for the Methods for Inspection, Test, and Standardization of Etched-Stem Liquid-in-Glass Thermometers (E 77).

The former Subcommittee 18 on Hydrometers has been re-established to continue the work on hydrometers. Mr. F. A. Lobisser, The George Ruehfel Co., Inc., has been appointed chairman of this subcommittee.

Subcommittee 19 on Glassware Laboratory Apparatus (J. J. Moran, chairman) is preparing up-to-date specifications for 100-ml bulb-type burets published in 1920 by the Manufacturing Chemists Assn.

Subcommittee 25 on Shear and Torsion Tests (R. W. Fenn, Jr., chairman) has approved the new test procedure, developed under the chairmanship of L. R. Lunsford, for determining the shear properties of adhesives for design purposes.

Section 1, under the chairmanship of R. B. Clapper, is continuing its efforts to prepare additional shear test procedures. Under study are the following: (1) a punch-shear test which determines the shear strength of sheet materials less than 0.250 in. in thickness in the short transverse direction; (2) a pin double-shear test; and (3) a sheet double-shear test. The latter test is also being studied as an elevated-temperature shear test for sheet by the Test Methods Panel of the Joint ASME-ASTM Committee. Liaison between Subcommittee 25 and the Test Methods Panel is being maintained.

Subcommittee 28 on Microscopy (T. G. Rochow, chairman) is sponsoring the Symposium on Resinographic Methods being held at the 1963 ASTM Annual Meeting in Atlantic City in June.

The Tentative Specifications and Methods of Test for Cover Slips for Use in Microscopy, appended to this report,[2] were prepared by the Section on Microscopes and Their Elements (O. W. Richards, chairman).

The American Standard B1.11-1958 for Microscope Objective Thread is recommended for approval as an ASTM standard.

The first addition to the Tentative Definitions of Terms Relating to Microscopy (E 175) prepared by Section 5 on Nomenclature (H. F. McMurdie, chairman) has been accepted by the Society. Additional definitions are now out to letter ballot of the subcommittee.

Section 6 on Embedding and Mounting Media (S. B. Newman, chairman) is preparing an extensive set of cards on optical properties of amorphous resins and monomers with references to the general literature.

Section 5 on Photomicrographical Materials (R. P. Loveland, chairman) and Section 7 on Electron Microscopy (F. F. Morehead, chairman) are working together on two mutual tasks: one of specification and the other of standardization. Section 7 has also begun work on problems involved in the measurement of resolving power (under the leadership of J. H. Reisner).

Section 8 on Interference Microscopy is being organized by its chairman, P. H. Bartels.

Subcommittee 29 on Microchemical Apparatus (A. Steyermark, chairman) prepared the Specifications for Apparatus for Microdetermination of Carbon and Hydrogen in Organic and Organo-Metallic Compounds (E 191), for Micropipets (E 193), and for Oxygen Combustion Flask (E 205). Specifications for vessels and apparatus used in the determination of alkoxyl and oxygen are now being considered.

Subcommittee 30 on Bend Test for Ductility (E. H. Enberg, chairman).—Under its new chairman this subcommittee is preparing a method for a semiguided bend test. It is expected that this will cover both bending around a pin of specified radius and bending without final application of the load in the immediate region of the bend but to a specified minimum radius.

Subcommittee 31 on Laboratory Balances (A. H. Corwin, chairman).—The program of work of this subcommittee is being reactivated under its new chairman. Drafts of proposed specifications will be prepared for consideration at the June meeting. It is expected that these will cover two types of balances in greatest demand at this time, namely, the 200-g equal arm balance and the 160-g substitution balance.

Subcommittee 32 on Thermocouples for Temperature Measurement (D. I. Finch, chairman).—The activities of this subcommittee have been transferred to Committee E-20 on Temperature Measurement.

Task Group B on Linear Thermal Expansion Test (H. E. Hagy, chairman) is completing a new proposed Tentative Method of Test for Linear Thermal Expansion of Rigid Solids with a Vitreous Silica Dilatometer. This method is intended for linear thermal expansion tests at −195 to +1000 C on rigid solids including metals, ceramics and refractories, glasses, rocks and minerals, inorganic cement, pastes and mortars, plastics, and wood.

This report has been submitted to letter ballot of the committee and its subcommittee chairmen, a total of 40 voting members; 34 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. J. McCoy,
Chairman.

P. J. Smith,
Secretary.

REPORT OF COMMITTEE E-2
ON
EMISSION SPECTROSCOPY*

Committee E-2 on Emission Spectroscopy held two meetings during the year: at the University of Maryland, College Park, Md., on June 21, 1962; and in Pittsburgh, Pa., on March 7, 1963. The Executive Subcommittee also met at these times as well as in Ottawa, Ontario, Canada, on Sept. 17, 1962.

The main activities of the committee were (1) concentration of efforts toward obtaining and testing new or improved methods and practices for inclusion in the planned Fourth Edition of the Methods for Emission Spectrochemical Analysis to be issued late 1963 or early 1964; and (2) the planning of a Symposium on X-Ray and Electron Probe Analysis (R. W. Smith, chairman) which is to be sponsored jointly with Committee E-4 on Metallography and is to be held June 27 and 28, 1963, at the Annual Meeting in Atlantic City, N. J.

The deadline for receipt of revisions and new material for the E-2 compilation was extended from March until the Annual Meeting in June, 1963. To facilitate printing and voting, a special supplement to this annual report will be issued and will contain at least 20 additional recommendations for changes or inclusions in the compilation.

The committee has 212 members, which is an increase of 7 since the last Annual Meeting. The official voting membership is 175.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee E-2 requests approval by the Society at the Annual Meeting of the following recommendations. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] All of the existing tentatives referred to appear in their present form in the 1960 Book of ASTM Methods for Chemical Analysis of Metals.

Suggested Methods to be Published as Information[2]

Suggested Methods for Spectrochemical Analysis of:

Gold-Antimony Alloys by the Solution-Residue Graphite-Spark Technique (SM 6-24),

Lead and Its Alloys by the Point-to-Plane, Argon Atmosphere, Spark Technique Using an Optical Emission Spectrometer (SM 6-25),

Gold Alloys by the Solution-Rotating Disk-Spark Technique (SM 6-26),

Indium and Its Alloys by the Point-to-Plane Condensed Arc Technique Using an Optical Emission Spectrometer (SM 6-27),

Gallium-Lead and Antimony-Lead Alloys by the Solution-Porous Cup-Spark Technique (SM 6-28),

Silver by the Powder - D-C Arc Technique (SM 6-29),

Stainless-Type Steels by the Point-to-Plane Spark Technique Using an Optical Emission Spectrometer (SM 9-23).

*** Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.**

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[2] The Suggested Methods appear in the compilation of *Methods for Emission Spectrochemical Analysis* (1963).

Stainless-Type 18/8 Steels by the Point-to-Plane Technique Using an Optical Emission Spectrometer (SM 9-24), and

Cement Raw Mix by the Lithium Tetraborate Fusion Technique Using a Recording X-Ray Spectrometer (SM 10–20).

Revision of Suggested Method

Suggested Method for Spectrochemical Analysis of Zirconium by the Silver Chloride Carrier—D-C Arc Technique (**SM 8-13**):

Section 1.—Delete the following elements and their concentration ranges: "Aluminum, Calcium, Lithium, Silicon, and Titanium."

Section 4(b).—Delete the following: "Aluminum Oxide (Al_2O_3), Calcium Carbonate ($CaCO_3$), Lithium Carbonate (Li_2CO_3), Silicon Dioxide (SiO_2), Titanium Dioxide (TiO_2)."

Table I.—Delete the following elements and their analytical lines and ranges: "Aluminum, Calcium, Lithium, Silicon (two places) and Titanium."

Section 8.—In Paragraph (*b*) delete the last line which reads "see Paragraph (*c*)."

Change Paragraph (*c*) to read "(*c*) *Exposure.*—It may be necessary to adjust the exposure parameter and repeat the procedure so that all of the concentration ranges may be adequately covered."

New Tentative

Tentative Method for Spectrochemical Analysis of Plain-Carbon and Low-Alloy Steel by the Rod-to-Rod Spark Technique, as appended hereto.[3]

Revision of Tentatives

Tentative Definitions of Terms and Symbols Relating to Emission Spectroscopy (E 135 – 60 T):

Revise as appended hereto.[3]

Tentative Recommended Practice for Designation of Shapes and Sizes of Preformed Graphite Electrodes (E 130 – 57 T):

Title.—Delete the word "Preformed."

New Section 2.—Add a new Section 2 entitled "References to These Practices in Standards" to read as follows, renumbering subsequent sections accordingly:

2. The inclusion of the following statement or equivalent in any ASTM spectrochemical method (preferably in the section on Reagents and Materials) shall constitute due notification that these recommended practices shall be followed:

Graphite Electrodes.—Use electrodes of high purity graphite which conform to shape C-1 or C-2 as given in the Recommended Practice for Designation of Shapes and Sizes of Graphite Electrodes (ASTM Designation: E 130).

Section 3.—Change present Section 3 to read as follows:

3. The dimensions of the electrodes are given in Fig. 1. Unless exceptions are shown, the following tolerances apply:

Angles, deg	± 0.5
Outside diameter, in.	± 0.002
Length, in.	± 0.016
Cavity, inside diameter, in.	± 0.003
Cavity depth, in.	± 0.003
Neck, outside diameter, in.	± 0.005
Neck location, in.	± 0.005
Neck length, in.	± 0.005
Concentricity, total indicated reading:	
Holes under 0.5 in. deep and undercuts, in.	0.005
Holes over 0.5 in. deep, in.	0.010
Rotating disks, inside diameter, in.	± 0.001
Rotating disks, thickness, in.	± 0.005
Boiler caps, inside diameter, in.	± 0.001
Porous cups, bottom thickness:	
Up to 1 in. length, in.	+ 0.002, −0.000
Over 1 in. length, in.	+ 0.003, −0.000

New Section 5.—Add a new Section 5 entitled "Definition" to read as follows:

5. *Preform.*—A descriptive word applied to a commercially produced spectroscopic electrode, which is purified after cutting or machining.

Figure 1.—Make the following changes in Figure 1:

C-1—Instead of listing the cone height of 0.312

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[3] The new and the revised tentatives appear in the *1964 Book of ASTM Standards*, Part 32.

in., list the included cone angle of 23 deg. Mark "0.062 in. flat or radius tip permitted."

C-4—Delete.

C-5—Change the tip radius from 0.062 to 0.06 in. Drawing should show a rounder tip.

C-7—Change the four 0.093 in. dimensions to 0.09 in. (post diameter, distance to flange, flange thickness and neck height).

C-8—Change the flange thickness and neck height to 0.09 in. from 0.093 in.

S-1—Instead of listing taper height of 0.125 in., list tip diameter as 0.086 in.

S-2—Change height from 0.625 to 0.62 in.; crater diameter from 0.156 to 0.16 in.; crater depth from 0.281 to 0.28 in.; and pedestal hole depth from 0.156 to 0.16 in.

S-3—Change height from 0.625 to 0.62 in.; crater depth from 0.281 to 0.28 in.; and pedestal hole depth from 0.156 to 0.16 in.

S-4—Change top-to-neck distance from 0.156 to 0.16 in.; cup diameter from 0.180 to 0.182 in.; neck height from 0.062 to 0.06 in.; and neck diameter from 0.121 to 0.12 in.

S-6—Change cup diameter from 0.156 to 0.16 in.

S-7—Delete.

S-8—Change cup diameter from 0.187 to 0.182 in.; and cup depth from 0.187 to 0.19 in.

S-9—Delete.

S-10—Delete.

S-11—Delete.

S-12—Change cup diameter from 0.187 to 0.182 in.; neck diameter from 0.187 (error) to 0.12 in.; and specify top-to-neck distance of 0.25 in. instead of crater-to-neck distance.

S-13—Delete the neck angle specification of 48 deg.; change cup depth from 0.187 to 0.19 in.; neck height from 0.031 to 0.03 in.; crater-to-base distance from 0.062 to 0.06 in.

S-14—Change cup diameter from 0.125 to 0.12 in.; add neck diameter of 0.12 in.; change cup depth from 0.187 to 0.19 in.; and specify distance top-to-neck of 0.25 instead of crater-to-neck of 0.062 in.

P-1—Change cup diameter from 0.180 to 0.18 in.; and crater depth from 0.218 to 0.187 in.

P-2—Show concave neck of 0.19 in. radius. (Similar to L3948 or UCP 104-L.)

PC-1—Change length from 1.0 to 0.88 in.; bottom diameter from 0.125 to 0.12 in.; and cup diameter from 0.156 to 0.16 in.

PC-2—Change cup diameter from 0.156 to 0.16 in., and length from 1.0 to 0.88 in.

D-1—Change thickness from 0.125 to 0.12 in.

D-2—Change thickness from 0.200 to 0.20 in.

D-3—Change centerpost diameter from 0.107 to 0.11 in.; add total height dimension of 0.20 in. instead of showing base-to-well distance of 0.197 in.; change tolerance on rim height 0.012 ± 0.003 in.; and diameter to rim from 0.4375 to 0.437 in.

D-4—Add. This is a mandrel for D-1 and D-2 with a length of 1.12 in.; mandrel top diameter 0.117 in. is 0.30 in. high, flares to 0.132 in. at bottom, and sits on 0.242 in. stock.

All electrodes showing a 118-deg angle, change to show a 30-deg drill point bottom. (This is equivalent.)

Tentatives Continued Without Revision

Tentative Recommended Practices for:

Describing and Specifying the Excitation Source in Emission Spectrochemical Analysis (**E 172 – 60 T**),

Photographic Processing in Spectrochemical Analysis (**E 115 – 59 T**),

Photographic Photometry in Spectrochemical Analysis (**E 116 – 59 T**), and

Spectrochemical Computations (**E 158 – 60 T**).

Tentative Methods for Spectrochemical Analysis of:

Zinc-Base Alloys and High-Grade Zinc by the Solution Residue - D-C Arc Technique (**E 27 – 59 T**),

Tin Alloys for Minor Constituents and Impurities (**E 51 – 59 T**),

Pig Lead by the Point-to-Plane Spark Technique (**E 117 – 59 T**), and

Aluminum and Aluminum-Base Alloys by the Point-to-Plane Spark Technique (**E 101 – 53 T**).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Apparatus and Equipment (A. W. Helz, chairman) is working on (1) description, specification, and testing of photographic emulsions; and (2) description and specifications of optical emission spectrometers, X-ray spectrometers, spectrographs, and microphotometers. The subcommittee plans to include in a supplement to this report a revision of the Suggested Practice for Describing and Specifying the Spectrograph (SM 1-3); a Suggested Practice for the Description and Specification of Photographic Emulsions; and a report on available photographic emulsions.

Subcommittee II on Fundamental Practices (J. E. Paterson, chairman) is working on (1) Unsolved Problems in Spectrochemical Analysis; and (2) evaluation and promulgation of the Suggested Practices for Use of Statistical Methods in Spectrochemical Analysis (SM 2-4), and for Establishing and Controlling Spectrochemical Analytical Curves (SM 2-6). Two new task groups have been established to write a practice for X-ray spectrometry and for use of spark sources. The committee plans to include in a later supplement a major revision of Unsolved Problems in Spectrochemical Analysis (Appendix I of 1960 Methods for Emission Spectrochemical Analysis). Because of the great interest in the Unsolved Problems Task Group (R. C. Barras, chairman), as demonstrated at the Pittsburgh Conference on March 4, 1963, plans have been initiated to hold a Symposium at Purdue in 1965 on this subject.

Material for the Index to the Literature on Spectrochemical Analysis,[4] covering the years 1955–1960, has been completed except for the subject index. The manuscript should be available shortly for printing. Work will then begin on the second Index to the Literature on X-ray Spectrographic Analysis.

Subcommittee III, Editorial (D. C. Spindler, chairman) has made additions (some revision) to the Definitions of Terms and Symbols Relating to Emission Spectroscopy (E 135) as appended to this report. The subcommittee also (1) has prepared a style guide for writing methods involving the use of spectrographs; (2) has made an editorial review of 29 methods; and (3) is continually seeking improved but simplified techniques to advance the editorial policy of the committee. The Publications Task Group chairman (P. B. Adams) under this subcommittee handles all the revisions, practices, and methods which will be included in the new compilation on Methods for Emission Spectrochemical Analysis.

Subcommittee IV on Electrodes, Pure Materials, Reagents, and Standards (W. R. Kennedy, chairman) has completed a major revision of the Recommended Practice for Designation of Shapes and Sizes of Preformed Graphite Electrodes (E 130) as included in this report. This revision has been necessary to specify and standardize new dimensions for electrodes and to eliminate electrodes not in use in Committee E-2 methods. The subcommittee is working on (1) availability of standard samples, reference samples, and high-purity materials for spectrochemical analysis; (2) physical and chemical properties of graphite and carbon used in spectrochemical analysis; (3) a general practice for sampling procedures and sample preparation techniques; (4) preparing a list of line pairs for various matrices; (5) obtaining information for publication on relative purity of elements used in the preparation of synthetic standards; and (6) a practice for gaseous atmosphere excitation. The subcommittee plans to complete by May, 1963, the manuscript of the "Report on Standard Samples, Reference Samples, and High-Purity Materials for Spectrochemical Analysis."[5]

Subcommittee V on Copper, Nickel, and High-Temperature Alloys (R. S. Kelly, chairman) has been divided into Sections as follows: A on Copper and Its Alloys (A. A. Di Leonardi, chairman); B on Nickel and Thermionic Nickel (D. O. Hill, chairman); and C on High-Temperature Alloys (D. E. Brown, chairman). Rhenium has been transferred to Subcommittee VIII. The subcommittee is working on (1) the testing of four suggested methods (SM 5-2, -9, -10, and -12) for acceptance as tentative; (2) cooperating with the National Bureau of Standards on homogeneity testing to make available primary standards for 42 copper-base alloys and for 23 high-temperature alloys. Two new suggested methods for analysis of high-temperature alloys, one by X-ray and the other by optical emission, are expected to be submitted in the near future. The subcommittee, however, does not plan any additional material for the summer supplement.

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[4] To be published as *ASTM STP No. 41-E.*

[5] To be published as *ASTM STP No. 58-E.*

Subcommittee VI on Lead, Tin, Zinc, Indium, Gallium, and Related Metals (J. F. Murphy, chairman) has been divided into Sections as follows: A on Zinc and Antimony (J. E. Van Dien, chairman); B on Lead and Tin (A. Goldblatt, Chairman); C on Bismuth and Cadmium (J. H. Hussey, chairman); D on Indium, Gallium, and Germanium (B. Fagan, chairman); E on Gold, Silver, and Platinum (C. E. Ingersoll, chairman); F on Sampling (T. P. Sheehy, chairman); and G on High-Purity Materials (A. Lincoln, chairman). This active subcommittee has prepared for publication the following suggested methods for spectrochemical analysis referred to earlier in this report: Gold-Antimony Alloys by the Solution Residue Spark Technique (SM 6-24); Lead and Its Alloys by the Point-to-Plane, Argon Atmosphere, Spark Technique Using an Optical Emission Spectrometer (SM 6-25); Gold Alloys by the Solution - Rotating Disk - Spark Technique (SM 6-26); Indium and Its Alloys by the Point-to-Plane Condensed Arc Technique Using an Optical Emission Spectrometer (SM 6-27); Gallium-Lead and Antimony-Lead Alloys by the Solution-Porous Cup-Spark Technique (SM 6-28); and Silver by the Powder—D-C Arc Technique (SM 6-29). Additional suggested methods also are being processed as well as test programs on four suggested methods (SM 6-9, -12, -15, and -18) which are planned for acceptance as tentatives. The subcommittee hopes to complete shortly the evaluation of test data and recommend two methods as tentative, and to recommend the publishing of seven new suggested methods now being processed.

Subcommittee VII on Aluminum, Magnesium, and Their Alloys (W. Tingle, chairman) has been working on processing new suggested methods and on acceptance of one suggested method as tentative by cooperative testing. The subcommittee plans to rewrite and recommend as tentative the Method for Aluminum and Its Alloys by the Point-to-Plane Spark and Intermittent Arc Techniques Using a Recording Spectrometer (SM 7-10). The subcommittee also plans to recommend the publication of two new suggested methods: Magnesium and Its Alloys by the Pin-to-Pin Spark Technique Using a Photoelectric Spectrometer, and Magnesium Alloys for Calcium by the Flame Photometer Technique.

Subcommittee VIII on Refractory Metals, Beryllium and Their Alloys (R. Alvarez, chairman) is cooperating with the National Bureau of Standards on the homogeneity testing and analysis for certification of six primary zirconium-base standard samples. This activity has resulted in the availability from the Bureau of two additional Zircaloy-2 standard samples. (Two zirconium and one Zircaloy-2 standards were made available previously.) Close liaison is being maintained between the activity of this subcommittee and similar activity in Committee E-3 on Chemical Analysis. Committee E-3 particularly has requested the development of a spectrochemical method for the analysis of beryllium for aluminum and magnesium. The subcommittee plans shortly to recommend publication of four new suggested methods: (1) Titanium Alloys by the Point-to-Plane Technique; (2) Molybdenum by the Powder–D-C Arc Technique; (3) Tungsten by the Powder–A-C and D-C Arc Techniques; and (4) Tantalum by the Silver Chloride-Graphite Carrier—D-C Arc Technique. Attempt also will be made to combine two current suggested methods (SM 8-12, -13) with a third unwritten method into a single method and promote this as tentative. A large amount of test data for the promulgation already is available.

Subcommittee IX on Ferrous Alloys, (J. F. Woodruff, chairman).—This subcommittee has been concentrating largely on test programs for acceptance of methods on ferrous alloys as tentative. The first ferrous method to be so recommended, based on extensive interlaboratory testing, is Plain-Carbon and Low-Alloy Steel by the Rod-to-Rod Spark Technique, appended to this report. Also, as referred to earlier, the subcommittee recommends publishing the two suggested methods on Stainless Type Steels by the Point-to-Plane Technique Using an Optical Emission Spectrometer (SM 9-23), and Stainless-Type 18/8 Steels by the Point-to-Plane Technique Using an Optical Emission Spectrometer (SM 9-24). Although these methods eventually may be combined into a single tentative, sufficient difference exists between them to publish them individually as suggested methods until such combination can be made. Some 20 active task groups comprise this subcommittee. Every effort is being made to complete the evaluation of test data and to recommend acceptance of at least four suggested methods as tentative. The four most probable are: (1) Plain-Carbon and Low-Alloy Steel by the Pellet Technique (SM 9-18); (2) Stainless-Type Steels by the Point-to-Plane Technique Using a Photoelectric Spectrometer (SM 9-11); (3) Determination of Acid-Soluble Aluminum in Low-Alloy Steels by the Solution Technique (SM 9-21); and Determination of Magnesium in Cast Iron by the Solution Technique (without prior publication as a suggested method).

Subcommittee X on Glass, Ceramics, Alkalies, and Cementitious Materials (W. M. Hazel, chairman) has recommended for publication the Suggested Method for Spectrochemical Analysis of Raw Mix Cements by the Lithium Tetraborate Fusion Technique Using an X-Ray Spectrometer (SM 10-20), referred to earlier in this report. This subcommittee also plans to recommend for publication two new suggested methods, one similar to SM 10-20 above, but for finished cements, and one on Soda-Lime Glass for Calcium Using an X-Ray Spectrometer. Important revisions of two suggested methods (SM 10-16 and -18) currently are being made and these hopefully also will appear in the summer supplement.

Subcommittee XI on Slags, Ores, and Other Miscellaneous Nonmetallic Materials (R. G. Russell, chairman) is working on (1) establishing the most suitable semi-quantitative method of spectrochemical analysis for testing and promulgation as tentative; (2) initiating a task group to provide a method for the analysis of samples in solution form; and (3) working on a test program for acceptance as tentative of the method on Blast Furnace and Steel Making Slags (SM 11-12). The subcommittee plans to recommend for publication the suggested method for Spectrochemical Analysis of Rocks and Minerals Using an X-Ray Spectrometer.

This report has been submitted to letter ballot of the committee, which consists of 172 members; 119 members returned their ballots, of whom 108 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

ROBERT E. MICHAELIS,
Chairman.

DAVID W. HENTHORN,
Secretary.

REPORT OF COMMITTEE E-3
ON
CHEMICAL ANALYSIS OF METALS*

Committee E-3 on Chemical Analysis of Metals held one meeting during the year: on June 27, 1962, in New York, N. Y., in conjunction with the Annual Meeting of the Society. The Advisory Committee, divisions, and task groups held individual sessions June 24 to June 28, 1962. The Advisory Committee also met in January at ASTM Headquarters in Philadelphia, Pa.

Methods for refractory-type metals and alloys received a great deal of attention during the year, as described under the activities of Division R.

The work load at task force level is high and every attempt is being made to increase participation by a greater number of people.

Initial plans for the formation of a new Division I, concerned with the analysis for gases in metals was initiated late in 1962, and an organizational meeting was held on March 5, 1963.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee E-3 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] All of the existing tentatives and standards referred to appear in their present form in the 1960 Book of ASTM Methods for Chemical Analysis of Metals.

Revision of Tentatives

Tentative Methods for Chemical Analysis of Zirconium and Zirconium-Base Alloys (E 146 – 62 T):

Revise as appended hereto.[2]

Tentative Methods of Test for Hydrogen Loss of Copper and Tungsten Powders (E 159 – 62 T):

Title.—Change to read "Tentative Method of Test for Hydrogen Loss of Copper, Tungsten, and Iron Powders."

Section 1.—Change to read as follows:

> 1. This method covers the determination of the hydrogen loss (see Section 5) of copper and iron powders in the range from 0.05 to 3.0 per cent and of tungsten powder in the range from 0.01 to 0.50 per cent. The results so obtained are an approximation of the oxygen content of the material in the absence of the interfering elements listed in Section 6(*a*) and (*b*).

Section 5.—Change to read "The loss in weight of the sample is determined on heating in hydrogen in a tube furnace at 875 C for copper and tungsten powders, or at 1150 C for iron powder."

Section 7.—Change Paragraphs (*b*) and (*c*) to read as follows:

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[2] The revised tentatives appear in the *1964 Book of ASTM Standards*, Part 32.

(*b*) *Combustion Tubes.*—Gastight, 30 in. long and 1 to 1.5 in. in diameter.

(*1*) *For Copper and Tungsten Powders.*—Use a silica, zircon, or sillimanite tube.

(*2*) *For Iron Powder.*—Use a zircon or sillimanite tube.

(*c*) *Combustion Boats.*—Approximately 3 in. long and $\frac{1}{4}$ in. wide. Condition the boat by heating in a hydrogen atmosphere to eliminate a possible error resulting from weight loss of the boat.

(*1*) *For Copper and Tungsten Powders.*—Use a fused aluminum oxide, quartz, or zircon boat.

(*2*) *For Iron Powder.*—Use a fused aluminum oxide or zircon boat.

Section 9.—Change Paragraph (*a*) and Note 3 to read as follows:

9. (*a*) Preheat the combustion tube to 875 ± 15 C for copper and tungsten powders, or to 1150 ± 15 C for iron powder, and flush thoroughly with nitrogen for 10 min (Note 3).

NOTE 3.—If nitrogen is not available, the following modification may be followed: Start a hydrogen flow of 3 to 5 bubbles per second through the cold combustion tube and ignite at the exit end after all of the air has been flushed out (minimum of 10 min). Raise the temperature to 875 ± 15 C for copper and tungsten powders, or to 1150 ± 15 C for iron powder, and insert the boat, prepared as directed in Paragraph (*b*), through the hydrogen flame. After 30 ± 2 min for copper and tungsten powders, or after 60 ± 2 min for iron powder, and with the hydrogen flowing, remove combustion tube from the furnace and allow to cool to below 35 C. Finish and calculate as described in Paragraph (*e*) and Section 10.

Change the first sentence of Paragraph (*d*) to read "After 30 ± 2 min for copper and tungsten powders, or 60 ± 2 min for iron powder, start the nitrogen flow, stop the hydrogen flow, and flush for at least 2 min."

Tentative Methods for Chemical Analysis of Reactor and Commercial Columbium (E 195 – 62 T):

Revise as appended hereto.[2]

Adoption of Tentatives as Standards Without Revision

Tentative Methods for Chemical Analysis of:

Ferroboron (**E 31 – 60 T**),
Magnesium and Magnesium-Base Alloys (**E 35 – 60 T**), and
Silver Brazing Alloys (**E 56 – 60 T**).

Adoption of Tentative as Standard With Revision

Tentative Methods for Chemical Analysis of Titanium and Titanium-Base Alloys (**E 120 – 60 T**):

Incorporate into Standard Methods E 120 – 60 all the procedures in the tentative revision except the two methods for aluminum (Sections 6 to 13, and Sections 77 to 83), which are to be continued as tentative.

Tentatives Continued Without Revision

Tentative Methods for Chemical Analysis of:

Steel, Cast Iron, Open-Hearth Iron, and Wrought Iron (**E 30 – 60 T**),
Aluminum and Aluminum-Base Alloys (**E 34 – 60 T**),
Special Brasses and Bronzes (**E 54 – 60 T**), and
Electronic Nickel (**E 107 – 60 T**).

Tentative Recommended Practice for:

Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals (**E 173 – 60 T**).

Tentative Methods of Test for:

Antimony in Copper and Copper-Base Alloys (**E 62 – 60 T**).
Oxygen in Molybdenum (**E 174 – 60 T**).

Tentative Photometric Methods for:

Determination of Phosphorus in High-Phosphorous Brazing Alloys (**E 156 – 56 T**).

Editorial Change

Standard Methods for Chemical Analysis of Titanium and Titanium-Base Alloys (**E 120 – 60**):

Section 79.—Delete Note 15 and renumber subsequent notes.

ACTIVITIES OF DIVISIONS

Editorial Subcommittee (Edna Jacobson, chairman) has completed the editorial revision of the zirconium and columbium metal methods. A procedure for processing methods, and a model method have been prepared for the guidance of division and task force chairmen. The inert-gas fusion method for oxygen in zirconium was processed by the subcommittee. The chairman of the editorial subcommittee is working in cooperation with Division F in rewriting the iron and steel methods.

Division R on Refractory Metals (G. Porter, chairman):

Columbium Metal Task Group (R. Fricioni, chairman)

Task force and editorial review on a photometric procedure for iron for inclusion in Methods E 195 have been completed as referred to earlier in the report. An ion exchange procedure for tantalum has successfully passed task group review and is being editorially revised to satisfy some objections. Sample buttons of varying composition will be prepared to test proposed methods for titanium, zirconium, and silicon. A revised carbon method has been proposed and will be given task group review in the coming year. The oxygen and hydrogen groups have been transferred to Division I.

Molybdenum Metal Task Force (R. H. Maurer, chairman)

To expedite method review in the absence of satisfactory solid samples, solution samples were analyzed for iron and nickel. Results in general were quite good. Work on a method for silicon has been in progress at the National Bureau of Standards and the chairman's laboratories. Fifty-pound bars of high-purity and "salted" molybdenum metal have been converted to chips at the Bureau and will be used for task force method review.

Tungsten Metal Task Group (H. B. Taylor, chairman)

The sub-groups for nitrogen, nickel, molybdenum, carbon, and iron have all obtained samples, made preliminary analyses, and then distributed these to the members along with methods. The first round results are available for carbon and nickel, and molybdenum is partially complete. Iron and nitrogen are in progress; some difficulties are reported with nitrogen.

Division S on Sampling (A. C. Holler, chairman):

Nonferrous Subcommittee (G. F. Donahue, chairman)

A proposed method for the sampling of copper and copper-base alloys was distributed to the technical committees interested in copper for their comments. Task groups have been appointed to prepare methods of sampling for cadmium, lead and lead-base alloys, and zinc and zinc-base alloys.

Miscellaneous Metals Subcommittee

Task groups are investigating the sampling of columbium.

Division G on General Analytical Methods (L. M. Melnick, chairman) is in the process of revising Recommended Practice E 50. Also, it is planned to revise Recommended Practice E 173 soon.

Division M on Miscellaneous Metals (S. Vigo, chairman):

Metallurgical Metal Powders, Group I

Group I has submitted a method for committee ballot on hydrogen loss of iron powder which will appear as a revision of

Method E 159, to cover a test for hydrogen loss of copper, tungsten, and iron powders. A method for chemical analysis of metal powders submitted earlier for editorial review is under review by the group to see if it covers all current needs. Based on requirements expressed by Subcommittee II-A of ASTM Committee B-9 on Metal Powders and Metal Powder Products, work is being initiated on evaluating and adapting to iron powder the appropriate sections of the Methods for Chemical Analysis of Steel, Cast Iron, Open-Hearth Iron, and Wrought Iron (E 30) dealing with total and graphitic carbon. It is planned to use the same specimens in a metallographic approach to determine combined carbon in order to correlate results. Group I also has plans to look into other areas of study suggested by Committee B-9.

Metallurgical Metal Powders, Group II

A photometric method proposed by the group for molybdenum in tungsten powder was evaluated and redrafted. In order to avoid duplication of effort with work on the same test under way in the Tungsten Task Force of Division R, the method and program were turned over to that task force for further evaluation on additional samples. Because of lack of interest in the test, work was discontinued on a method for nonvolatile material in tungsten powder. Group II, having discharged its current assignments, is now inactive.

Titanium

The task group has been conducting cooperative testing of three proposed methods for copper and one for zirconium in titanium alloys. A proposed revision of the method for vanadium in titanium (E 120) has also been under cooperative test. A method for hydrogen by hot extraction has also been undergoing revision as a result of a negative ballot; the objective of the revision is to generalize the description of the method in order to have it apply to existing apparatus available from various sources.

Zirconium-Hafnium

A revision of the iodate titration (volumetric) method for tin in the Methods for Chemical Analysis of Zirconium and Zirconium-Base Alloys (E 146) has been submitted for committee ballot. A method for oxygen by inert-gas fusion has been evaluated and submitted for committee ballot as an addition to Method E 146. The task force has completed cooperative work with the National Bureau of Standards on five zirconium and zircaloy standards and is planning additional work on two new standards. In the photometric method for silicon, certain interferences have been reviewed and will be further investigated. Plans have been made to initiate cooperative testing of methods for boron and for uranium-235 enrichment in zirconium. Also, a polarographic method for tin will receive additional cooperative testing, as interest in this method is being retained. Work on carbon in zirconium has been reactivated to extend the scope below 200 ppm of carbon. Work on cobalt, molybdenum, columbium, and tantalum in zirconium is not considered needed at the present time.

Some work was done on cooperative testing of specially-prepared hafnium metal samples (using the analytical methods developed for zirconium metal) for iron, nitrogen, hydrogen, uranium, silicon, tungsten, copper, and titanium. It appeared that fairly acceptable results were obtained for all these elements except copper and titanium; the disagreements in all cases were attributable to segregation and the difficulty of preparing homogenous ingots. It is felt that the zirconium methods are applicable and can be extended to cover the aforementioned eight elements in hafnium, as well as chromium, tin, nickel, manganese, and aluminum. However, it is planned to test the methods for oxygen and carbon, as developed for zirconium, on samples prepared from hafnium sponge, since some changes in the conditions of test are considered to be necessary.

Uranium

Division M is waiting for publication of the Atomic Energy Commission analytical methods referenced in specifications on uranium under the jurisdiction of ASTM Committee B-2 on Nonferrous Metals and Alloys in order to study how it can cooperate in establishing Committee E-3 methods.

Division N on Nonferrous Metals (R. G. Ernst, chairman).—Activities in the various task groups of the division have been as follows:

Revision of Copper-Base Methods (L. W. Anderson, chairman)

A new procedure for the volumetric determination of iron by titration with ceric sulfate has been tested and is ready for balloting. Practical considerations appear to work against a consolidation of all copper-base methods under one designation, but a certain amount of rearrangement may be possible when the editorial revision is completed.

Revision and Consolidation of Methods for Copper-Nickel and Nickel-Copper Alloys (O. P. Case, chairman)

Not all the changes under consideration have been accepted, particularly the deletion of certain procedures. An attempt will be made to reach agreement on these items before any editorial revision is undertaken.

Revision of Methods for Nickel Metal, Including Electronic-Grade Nickel (A. D. Middleton, chairman)

Supporting data are being accumulated from task group members on several new procedures to be incorporated in the revision of the methods for the analysis of nickel. It is planned to incorporate the methods for the analysis of electronic-grade nickel into the revised group of methods. A few procedures are still being studied before a final selection is made of the ones to be tested cooperatively.

Revision of Methods for Aluminum Base Alloys (G. B. Wengert, chairman)

A thorough consideration of these methods has indicated the need for some changes, which will require cooperative testing, as well as considerable editorial changes, deletions, limitations of scope, etc. Since these methods have not been completely revised for a number of years, an extensive modification is expected.

Revision of Methods for Zinc and Zinc-Base Alloys (K. C. Braun, chairman)

New methods for the analysis of zinc alloys, employing the most recent techniques, are being considered in connection with the proposed revisions of the methods for the analysis of zinc and zinc alloys. Determination of trace amounts of aluminum in spelter and the analysis of zinc anodes for cathodic protection will be added to the revised procedures.

Review and Revision of Methods for White Metal Alloys and Solder (J. W. Claypool, chairman)

Some opposition has developed to the proposal for the consolidation of the methods for these two classes of materials. The task group will attempt to resolve the differing viewpoints. A method for the determination of silver has been written and tested and a method for the determination of traces of aluminum is under consideration.

Analysis of Silver Brazing Alloys (G. F. Donahue, chairman)

Four samples, containing high and low levels of lead, tin, iron, and nickel in brazing alloys of both high and low cadmium content have been prepared for use in the testing of proposed procedures for the determination of these elements in silver brazing alloys. It is believed that the methods for the determination of lead, iron, and nickel in copper-base alloys will apply to these samples with little, if any, modification. A method for the determination of tin at low levels will require further study.

Analysis of Beryllium (S. Kallmann, chairman)

This group, under its present chairman, has been reactivated, with especial emphasis to be directed to the analysis of beryllium metal rather than the oxide. Several procedures have been suggested for study by the group. A problem has arisen concerning the specification, which calls for a maximum content of beryllium oxide. Attempts are being made to determine whether a total oxygen content can be specified instead.

Analysis of Lithium Metal (P. F. Collins, chairman)

Interlaboratory testing of solutions of lithium salts were made by several of the cooperating laboratories, using their own procedures for the determination of sodium, potassium, iron, and chloride. Based on these results, a method for chloride based on a turbidimetric measurement is being written, and two methods for iron are being studied. Sodium and potassium are best determined by flame photometry and properly come under the jurisdiction of Committee E-2 on Emission Spectroscopy. Liaison will be worked out.

Analysis of Cadmium Metal (R. G. Ernst, chairman)

This newly activated group held an organization meeting, selected a group of methods for testing, and discussed procedures for determination of all the elements included in the proposed specification for cadmium. Initial effort will be directed toward the determination of copper, iron, lead, and zinc, with thallium and bismuth scheduled for early consideration. Samples for cooperative testing have been made available.

Division I on Gases in Metals (T. D. McKinley, chairman). Seven task groups are in various stages of operation as enumerated below:

Task Force on Oxygen in Columbium (C. H. Albright, chairman)

Work with vacuum fusion technique in range 50 to 300 ppm oxygen has just been concluded in a very satisfactory manner. Work with inert gas fusion technique continues at the lower end of the range and should be concluded in 1963.

Task Force on Oxygen in Zirconium (S. Kallmann, chairman)

Work to give a satisfactory tentative method (under Division M) was concluded. The task force is continuing work at lower levels which should be concluded in 1963.

Task Force on Oxygen in Molybdenum and Tungsten (W. F. Harris, chairman)

Specimens are about ready for distribution in the task group.

Task Force on Hydrogen in Steel (J. F. Martin, chairman)

Two specimens of austenitic stainless steel are being screened prior to distribution.

Task Force on Oxygen in Tantalum (D. M. Mortimore, chairman)

The task group is in a formative stage.

Task Group on Oxygen in Beryllium (S. Kallmann, chairman)

Activation awaits interpretation of specifications.

Task Group on Oxygen in Chromium (F. P. Valente, chairman)

Division F on Ferrous Metals (M. D. Cooper, chairman) has held two meetings: at New York City on June 25, 1962, and at Pittsburgh, on March 5, 1963. Task groups are pursuing projects as follows:

Hydrogen in Steel

This task group was transferred to Division I by action of the Advisory Committee on January 28, 1963.

Low Carbon (less than 0.01 per cent) in Steel

The conductometric method with standard samples for calibration has been evaluated; the use of potassium acid phthalate as the primary standard is being studied.

Nitrogen in Steel

A method for the determination of total nitrogen in carbon steels is practically completed.

High Cobalt in Steels and Other Alloys

The ion exchange-titrimetric method appears to require only minor modification before gaining acceptance by the task group.

Low Sulfur (less than 0.01 per cent) in Steel

Data have been obtained by a combustion method with standard samples used for calibration. The method will be processed with the existing method when it has been rewritten and evaluated. An umpire method still is being sought.

Aluminum (up to 0.1 per cent) in Steel

This task group was appointed at the June, 1962, meeting; evaluation of a photometric method will begin as soon as special heats providing aluminum in the appropriate concentration range can be prepared.

Aluminum (0.1 to 5.0 per cent) in Steel

The 8-hydroxyquinoline method, which has been under investigation for some time, has given good results when applied with either the gravimetric or titrimetric procedure. Further evaluation, however, may be limited to the gravimetric procedure.

Ferrocolumbium

The anion exchange methods for columbium, tantalum, and titanium will be submitted in the group for balloting; development work is continuing on methods for manganese, silicon, tin, phosphorus, aluminum, and carbon.

Boron in Ferro Alloys

Methods and samples have been submitted to the task group.

Aluminum in Ferro Alloys

Three methods are being considered by the task group; when one is selected, initial tests will be carried out with ferrosilicon. Modifications will be made as required for other alloys.

Arsenic in Ferro Alloys

Preliminary evaluation of a distillation-photometric method for arsenic in ferrosilicon, high-carbon ferrochrome, and silicomanganese indicated a need for some revisions.

Lead in Ferro Alloys

A dithizone photometric method is being evaluated for lead in high-carbon and low-carbon ferrochrome, ferromanganese, and ferromolybdenum.

Tin in Ferro Alloys

Preliminary results by an extraction-photometric method for tin in ferrosilicon, ferromanganese, ferrocolumbium, and low-carbon ferrochromium are encouraging.

Quality of Sodium Bismuthate

An exhaustive study of currently available sodium bismuthate has failed thus far to reveal the reason for its failure to yield complete oxidation of manganese.

Manganese Metal as a Primary Standard

The feasibility of establishing the assay of commercially available manganese for use as a primary standard is being studied; establishing values for gases is of special interest. The use of sulfurous acid to reduce the contribution of oxides on the surface of the metal shows promise, but the stability in this regard after cleaning remains to be established. Metal in the form of 150 to 200-mg lumps is being evaluated; the total impurities in one lot, including gases, were shown to amount to only 0.12 per cent.

Rewriting Existing Methods

Revisions of methods in accordance with the Style Manual are nearing completion. Similar treatment of additional methods is in progress. As this phase of the work is accomplished, task forces will be appointed to evaluate and to test the revised methods with sufficient thoroughness to permit the inclusion of a statement related to the precision of the method. Methods soon to be evaluated are: (1) carbon by the direct-combustion gravimetric method, (2) phosphorus by the alkalimetric method, (3) phosphorus by the molybdenum blue (photometric) method, (4) manganese by the periodate (photometric) method, and (5) manganese by the persulfate-arsenite method.

This report has been submitted to letter ballot of the committee, which consists of 127 members; 78 members returned their ballots, of whom 75 have voted affirmatively, and 0 negatively.

Respectfully submitted on behalf of the committee,

ARBA THOMAS,
Chairman.

H. KIRTCHIK,
Secretary

REPORT OF COMMITTEE E-4
ON
METALLOGRAPHY*

Committee E-4 on Metallography and several subcommittees met in conjunction with the Annual Meeting of the Society, June 24 to 27, 1962, in New York, N. Y. In addition, there were subcommittee and task group meetings in Pittsburgh, Pa. on Nov. 7 to 9, 1962.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1962 Annual Meeting, Committee E-4 presented to the Society through the Administrative Committee on Standards the following recommendation:

Revision of Tentative Recommended Practice for:

Determining the Inclusion Content of Steel (E 45 - 60 T).

The recommendation was accepted by the Standards Committee on January 4, 1963, and the revised recommended practice appears in the 1962 Supplement to Book of ASTM Standards, Part 3.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee E-4 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] All of the existing tentatives and standards referred to appear in their present form in the 1961 Book of ASTM Standards, Part 3.

Adoption of Tentatives as Standard Without Revision

Tentative Methods for:

Preparing Quantitative Pole Figures of Metals (**E 81 - 54 T**),

Determining the Orientation of a Metal Crystal (**E 82 - 49 T**), and

Assigning Phase Designations in Metallic Systems (**E 157 - 61 T**).

Tentative Definitions of Terms Relating to:

Metallography (**E 7 - 55 T**).

Tentative Recommended Practices for:

Thermal Analysis of Metals and Alloys (**E 14 - 51 T**),

Determining the Inclusion Content of Steel (**E 45 - 62 T**),

Dilatometric Analysis of Metallic Materials (**E 80 - 49 T**), and

Resistometric (or EMF) Analysis of Metallic Materials (**E 189 - 61 T**), with an editorial change in title to read: "Tentative Recommended Practice for Determining Temperature—Electrical Resistance Characteristics (EMF) of Metallic Materials."

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Revision of Standard,
Immediate Adoption

(requires nine-tenths affirmative vote)

Standard Methods for Estimating the Average Grain Size of Metals (E **112–61**):

Revise as appended hereto.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Selection and Preparation of Samples (W. D. Forgeng, chairman) is preparing an extensive revision of etching procedures and etchants to bring the Methods of Preparation of Metallographic Specimens (E 3) in line with present practice.

Subcommittee III on Nomenclature (W. L. Fink, chairman) has prepared a recommended procedure for presenting phase diagrams.

Subcommittee IV on Photography (G. A. Ellinger, chairman) collected and arranged the Photomicrographic Exhibit at the 1962 Annual Meeting. The exhibit proved to be an outstanding success. The subcommittee is already busily preparing for the 1964 exhibit.

Subcommittee VI on X-Ray Methods (R. K. Scott, chairman) has under final review the replacement for the withdrawn Recommended Practice E 43 on powder diffraction methods. Due to the magnitude of this document, it will be described only briefly in the Book of Standards. The complete document will be published separately.

The subcommittee is continuing the study of methods for the indexing of electron diffraction data.

Subcommittee VIII on Grain Size has finally attained full agreement on the universal application of Methods E 112. Agreement was facilitated by the following actions:

(1) Approval by Committee E-4 and Committee B-5 on Copper and Copper Alloys, of an extract of Methods E 112. The extract, "Estimating the Average Grain Size of Wrought Copper and Copper-Base Alloys," makes Methods E 112 applicable to materials under the jurisdiction of Committee B-5 and paves the way for the withdrawal of the Methods for Estimating the Average Grain Size of Wrought Copper and Copper-Base Alloys (E 79).

(2) Committee E-4 approval of changes in Methods E 112 to make the latter consistent with the extract.

With the withdrawal of Methods E 79 comes the achievement of a goal for which Committee E-4 and other committees have worked for over 25 years to attain—the reduction of grain size measurement to a common base (a problem in geometry) and the elimination of materials-directed implications.

Additionally, Subcommittee VIII is collaborating with Subcommittee XIV and with Subcommittee VII of Committee A-3 on Cast Iron on the subject of comparison charts for graphite in cast iron.

Subcommittee IX on Inclusions (Samuel Epstein, chairman) completed the revision of Recommended Practice E 45 which was accepted by the Administrative Committee on Standards. The latest text extends the practice to the rating of inclusions in high-quality vacuum melted steels.

Subcommittee XI on Electron Microstructure of Metals (J. R. Mihalisin, chairman) is continuing its technical symposia on advanced techniques in electron microscopy and electron microprobe analysis. To their list of Special Technical Publications, there has recently been added another, "Advances in Electron Metallography and Electron-

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[2] The revised standard appears in the 1963 Supplement to *Book of ASTM Standards*, Part 3.

Probe Microanalysis," *ASTM STP No. 317*.

The subcommittee is conducting interlaboratory tests on electron microprobe analysis and is also cooperating in a joint program with Committee A-10 on Corrosion-Resistant Alloys on the study of type 316 stainless steel.

Subcommittee XIV on Quantitative Metallography (G. E. Pellissier, chairman) has held several meetings, the first of which consisted of a general survey of methods. At present, task groups are correlating conventional methods and computer - scanning methods.

The subcommittee is collaborating with Subcommittee VIII and with Subcommittee VII of Committee A-3 in the correction of the old A 247 flake graphite comparison charts and in the preparation of a new series of charts for nodular irons.

This report has been submitted to letter ballot of the committee, which consists of 124 members; 108 members returned their ballots, of whom 98 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

L. L. WYMAN,
Chairman.

M. R. NORTON,
Secretary.

REPORT OF COMMITTEE E-5

ON

FIRE TESTS OF MATERIALS AND CONSTRUCTION*

Committee E-5 on Fire Tests of Materials and Construction held one meeting during the year: on Feb. 8, 1963, in Montreal, Canada.

The committee consists of 76 members, of whom 37 are classified as producers, 6 as consumers, 28 as general interest members, with 3 honorary and 2 consulting members.

Recommendations for Society Action at Annual Meeting

Committee E-5 requests approval by the Society at the Annual Meeting of the following recommendations. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] The existing tentatives and standards referred to appear in their present form in the 1961 Book of Standards, Part 5.

Tentatives Continued Without Revision

Tentative Method of Tests for Determining Noncombustibility of Elementary Materials (**E 136 – 59 T**), and

Tentative Methods of Fire Tests of Window Assemblies (**E 163 – 60 T**).

Tentative Revision of Standard

Standard Methods of Fire Tests of Building Construction and Materials (**E 119 – 61**):

Section 6.—Add a new Paragraph (*c*) to read as follows:

> (*c*) When it is desired to include a built-up roof covering, the test specimen shall have a roof covering of 3-ply, 15-lb type felt and not in excess of 120 lb per square of hot mopping asphalt without gravel surfacing. Tests of assemblies with this covering do not preclude the field use of other built-up roof coverings.

American Standards

The committee, in cooperation with the Underwriters' Laboratories, recommends that the following methods be referred to the American Standards Association for approval as American Standard:

Standard Method of Test for Surface Burning Characteristics of Building Materials (**E 84 – 61**), and

Tentative Methods of Fire Tests of Window Assemblies (**E 163 – 60 T**).

Activities of Subcommittees

Subcommittee I on Fire Tests of Material and Construction (A. J. Steiner, chairman).—Task groups are now engaged in: determining the fire resistance rating of loaded beams; clarifying the end point criteria of ignition of cotton waste; reviewing conditioning requirements for test specimens; and reviewing the feasibility of adopting a standard test report form for Methods E 119.

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

A joint task group has been set up with Committee C-20 on Acoustical Materials to clarify ceiling tests as described in Methods E 119.

Subcommittee IV on Fire Tests of Acoustical and Similar Finishes (C. H. Yuill, chairman).—Task groups are engaged in: determination of methods of measuring smoke emission; a study of the hazards of combustion; and preparation of a new test standard for surface finishes.

A group has been established to prepare a commentary on the techniques of conducting tests under the committee's jurisdiction for inclusion as an appendix to the test methods.

Subcommittee V on Nomenclature and Definitions (J. V. Ryan, chairman) is studying new definitions for addition to Definitions E 176. A task group has been appointed to study the feasibility of developing a calorimetric method for defining low combustibility.

Subcommittee X on Research (A. F. Robertson, chairman) is sponsoring a symposium on the effects of moisture on fire tests of building materials at the 1964 Annual Meeting.

This report has been submitted to letter ballot of the committee, which consists of 72 voting members; 68 members returned their ballots, of whom 66 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

GORDON W. SHORTER,
Chairman.

IRWIN A. BENJAMIN,
Secretary.

REPORT OF COMMITTEE E-6

ON

METHODS OF TESTING BUILDING CONSTRUCTIONS*

Committee E-6 on Methods of Testing Building Constructions held two meetings during the year: in New York, N. Y., on June 26, 1962; and in Montreal, Canada, on Feb. 5, 1963.

The committee consists of 102 members, of whom 96 are voting members; 47 are classified as producers, 8 as consumers, and 41 as general interest members. There are 6 consulting members.

As a result of a change in the by-laws providing for a second vice-chairman, the office was filled by the appointment of E. C. Shuman by the Executive Committee.

Recommendations for Society Action at Annual Meeting

Tentatives Continued Without Revision

Tentative Recommended Practice for:

Laboratory Measurement of Airborne Sound Transmission Loss of Building Floors and Walls (**E 90 - 61 T**).

Tentative Methods of Test for:

Bond Strength of Mortar to Masonry Units (**E 149 - 59 T**), and

Materials for Use as Vapor Barriers Under Concrete Slabs and as Ground Cover in Crawl Spaces (**E 154 - 60 T**).

Activities of Subcommittees

Subcommittee I on Segments of Wall, Floor, and Roof Construction (J. A. Liska, chairman) reviewed comments from users of the wet racking test method but no changes were recommended.

The editorial revision of Methods E 72 by the subcommittee has been completed and will be submitted to the Society.

Subcommittee II on Durability (E. C. Shuman, chairman).—The second draft of the Recommended Practice for Building Construction to Enhance Durability Against Water Damage was reviewed and a third draft will be prepared in light of the comments and sent out for ballot of the subcommittee.

Subcommittee III on Sound Transmission (R. K. Cook, chairman) was recently designated to serve also as Subcommittee VI of Committee C-20 on Acoustical Materials. The first meeting under the new dual arrangement was held in Philadelphia on Oct. 25, 1962. The principal activity concerns two projects: (1) development of a laboratory method for measuring impact sound transmission, and (2) development of a field method for measuring airborne sound transmission loss. A round-robin test program involving four laboratories is currently in progress on the impact sound study. Other matters dealt with during the meeting concerned acoustical terminology in ASTM standards, sound transmission through suspended ceilings, and laboratory installation of operable and movable partitions for sound transmission measurements.

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

Subcommittee IV on Unit Masonry (C. B. Monk, chairman) is continuing work on the development of a test method for shear.

Subcommittee V on Trusses, Beams, and Columns (Hugh Angleton, chairman).—Under new chairmanship the subcommittee accepted a report of a task group that work should be carried out on minor revisions of Methods E 73 for the purpose of classification of the methods and further study of any major revisions pending the outcome of various research projects now under way. Liaison was established between Subcommittee V of Committee E-6 and the subcommittee of Committee D-7 on Wood concerned with test methods of timber fastenings.

Subcommittee VI on Load Tests of Completed Structures (W. R. Schriever, chairman) met twice during the year: in Dallas, Tex., on Feb. 5, 1962, and in New York, N. Y. during the Annual Meeting on June 25, 1962. Among the various tests on walls the subject of a door slamming test for the evaluation of light partitions incorporating a door was considered to deserve first priority. A task group was appointed to prepare a draft method for such a test.

Subcommittee VII on Vapor Barriers Beneath Concrete Slabs on the Ground (L. O. Anderson, chairman).—The study of reports on use of the Tentative Methods of Testing Materials for Use as Vapor Barriers under Concrete Slabs and as Ground Cover in Crawl Spaces (E 154) was continued with emphasis on abrasion versus puncture tests, resistance to decay tests, and tests for resistance to deterioration from petroleum vehicle for soil poisons. Consideration was given for the need of a flame spread test.

Subcommittee VIII on Windows (L. M. Dunn, chairman) prepared drafts of proposed methods for water leakage and structural strength under the influence of wind loads, and continued its consideration of a draft of a proposed method for air leakage. The drafts on air leakage and structural strength were submitted to letter ballot among the subcommittee members. Further work on all three drafts is being carried on.

Subcommittee IX on Curtain Walls (C. L. Pettibone, chairman) reports progress in the development of test methods for air infiltration, water infiltration, thermal transfer, and wind loading. Liaison has been established with Subcommittee VIII and with Committees C-16 on Thermal Insulating Materials and C-19 on Structural Sandwich Constructions because of the similarity of test methods being developed and assemblies that may be tested alone or in combination such as walls and windows. Reports have been made on test methods presently used by industry and a study of them is being conducted in order that they may be considered as possible ASTM test methods. The subcommittee is also working on nomenclature for use in material concerning curtain walls.

This report has been submitted to letter ballot of the committee, which consists of 107 voting members; 71 members returned their ballots, of whom 68 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

R. F. LEGGET,
Chairman.

J. P. THOMPSON,
Secretary.

REPORT OF COMMITTEE E-7
ON
NONDESTRUCTIVE TESTING*

Committee E-7 on Nondestructive Testing met in New York, N. Y. on June 25, 1962, in conjunction with the Annual Meeting of the Society. The Executive Council, seven subcommittees, and task groups met at this time. Meetings were also held in Montreal, Canada, Feb. 7, 1963, during Committee Week.

The new officers elected at the 1962 Annual Meeting for a two-year term of office are as follows:

Chairman, A. Gobus.
First Vice-Chairman, G. R. Forrer.
Second Vice-Chairman, C. Jenni.
Secretary, W. E. Havercroft.
Assistant Secretary, R. W. McClung.

J. Delisa was appointed chairman of Subcommittee I on Radiographic Recommended Practice, to replace G. R. Forrer. J. W. Allen has resigned as chairman of Subcommittee VII on Electromagnetic Methods.

Recommendations Accepted by the Administrative Committee on Standards

Subsequent to the 1962 Annual Meeting, Committee E-7 presented to the Society through the Administrative Committee on Standards the following recommendations which were accepted on the dates indicated:

New Tentative Reference Radiographs of Investment Steel Castings for Aerospace Applications (E 192 - 62 T) (Accepted July 23, 1962)

Revision of Tentatives:

Recommended Practice for Radiographic Testing (E 94 - 52 T) (Accepted Oct. 17, 1962)

Method of Ultrasonic Contact Inspection of Weldments (E 164 - 60 T) (Accepted Sept. 11, 1962)

The new Recommended Practice and the revised Reference Radiographs appear in the 1962 Supplement to Book of ASTM Standards, Part 3. The revised Method E 164 has been issued as a separate reprint.

Recommendations for Society Action at Annual Meeting

Committee E-7 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] All of the existing tentatives referred to in this report appear in their present form in the 1961 Book of ASTM Standards, Part 3.

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

New Tentatives

as appended hereto.[2]

Tentative Method for:

Ultrasonic Inspection of Metal Pipe and Tubing for Longitudinal Discontinuities.

Tentative Recommended Practices for:

Immersed Ultrasonic Testing by the Reflection Method Using Pulsed Longitudinal Waves,
Electromagnetic Testing of Seamless Aluminum Alloy Tube, and
Measuring Coating Thickness by Magnetic or Electromagnetic Methods.

Adoption of Tentatives as Standard Without Revision

Tentative Methods for:

Dry Powder Magnetic Particle Inspection (**E 109 - 57 T**),
Wet Magnetic Particle Inspection (**E 138 - 58 T**), and
Liquid Penetrant Inspection (**E 165 - 60 T**).

Tentative Reference Radiographs for:

Steel Welds (**E 99 - 55 T**).

Tentative Recommended Practice for:

Ultrasonic Testing by the Reflection Method Using Pulsed Longitudinal Waves Induced by Direct Contact. (**E 114 - 55 T**).

Tentative Reference Photographs for:

Magnetic Particle Indications on Ferrous Castings (**E 125 - 56 T**).

Tentatives Continued Without Revision

Tentative Method for:

Controlling Quality of Radiographic Testing (**E 142 - 59 T**).

Tentative Recommended Practices for:

Ultrasonic Testing by the Resonance Method (**E 113 - 55 T**), and
Fabricating and Checking Aluminum Alloy Ultrasonic Standard Reference Blocks (**E 127 - 61 T**).

Tentative:

Industrial Radiographic Terminology for Use in Radiographic Inspection of Castings and Weldments (**E 52 - 49 T**), and
Reference Radiographs for Inspection of Aluminum and Magnesium Castings (Series II) (**E 155 - 60 T**).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Radiographic Recommended Practice (J. Delisa, chairman) completed the revision of Recommended Practice E 94 which was accepted by the Standards Committee on Oct. 17, 1962. The task group will remain active to consider further revisions. Work on the radiographic sensitivity of thin metal specimens (up to $\frac{1}{16}$ in.) will be continued when a suitable penetrameter is obtained for these materials. The subcommittee is preparing a document illustrating for general guidance and education, the variations in appearance of a radiographic image if parameters such as radiation energy, standard thickness, and recording media are changed. Radiographs of a weld will be used separately or as a composite group of radiographs $2\frac{1}{2} \times 3$ in. to illustrate the weld and penetrameter image as the above parameters are changed.

Subcommittee II on Reference Radiographs (S. Goldspiel, chairman) has completed the tentative Reference Radiographs for Heavy-Walled Steel Castings (2 to $4\frac{1}{2}$ in.) (E 186) which was accepted by the Standards Committee on April 25, 1962.

The Tentative Reference Radiographs of Investment Steel Castings for Aerospace Applications (E 192) was accepted by the Standards Committee on July 23, 1962.

The Noah A. Kahn Memorial Award prepared by this subcommittee will likely be presented to the first recipients of the award during 1963.

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[2] The new tentatives appear in the 1963 Supplement to *Book of ASTM Standards*, Part 3.

The preparation of reference radiographs for steel thicknesses of 4½ to 12 in. is being continued by the task group.

Subcommittee III on Magnetic Particle and Penetrants (H. Migel, chairman):

Task Group A is preparing reference photographs for penetrant inspection. These photographs will be classified according to the appearance of the indication rather than to the type or cause of discontinuity which they indicate.

Task Group B is preparing a method of evaluating penetrant systems. A survey is being made of work done in this field. Work is continuing on the preparation of reference photographs for wet magnetic particle inspection and a task group is active in the preparation of a glossary of terms used in magnetic particle and penetrant inspection.

Subcommittee IV on Nomenclature (G. Corney, chairman) is responsible for the gathering of terms and definitions pertinent to the field of nondestructive testing. Glossaries submitted by other subcommittees of Committee E-7 will be edited and documented.

Subcommittee V on Penetrameters (E. L. Criscuolo, chairman).—A task group is studying new penetrameter designs suitable for use with very thin sections. Standard design penetrameters do not seem suitable. A comparison of the different types of penetrameters used in the United States and abroad was prepared by the subcommittee and reported in the ASTM BULLETIN. Recommendations have been made on the identification of penetrameters.

Subcommittee VI on Ultrasonic Testing (R. B. Moyer, chairman).—Section J of this subcommittee has completed the new tentative method for Ultrasonic Inspection of Tubing and Pipe as appended to this report. Similarly Section F has completed the new tentative Recommended Practice for Immersed Ultrasonic Testing which is appended to this report. The subcommittee is also working on the ultrasonic method of weld testing. An organization structure and operating procedure for the subcommittee will be available soon.

Subcommittee VII on Electromagnetic Methods (new chairman being considered) has prepared a document on the Electromagnetic Testing of Nonmagnetic Tube which is now ready for approval by Committee E-7. The subcommittee has a task group preparing a glossary of terms used in electromagnetic testing and a group collecting operating procedures and reference standards for testing of magnetic tubing.

This report has been submitted to letter ballot of the committee, which consists of 167 members; 129 members returned their ballots, of whom 123 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

A. GOBUS,
Chairman.

WILLIAM E. HAVERCROFT,
Secretary.

REPORT OF COMMITTEE E-8
ON
NOMENCLATURE AND DEFINITIONS

Committee E-8 on Nomenclature and Definitions held two meetings during the year: in New York, N. Y., on June 28, 1962, in conjunction with the Annual Meeting, and in Philadelphia, Pa., on Jan. 10, 1963.

One of the activities during the past year was a review of the preliminary Word List for Glossary of ASTM Definitions. Consideration was given to segregating from the main glossary all terms that are of general scientific interest. Three task groups of members and representatives of Committee E-8 were formed, each to review one third of the Word List, with the purpose of determining what the general terms are.

The committee also reviewed new definitions received from several committees during the year and made comments.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1962 Annual Meeting, Committee E-8 presented to the Society through the Administrative Committee on Standards the following recommendations which were accepted on the dates indicated:

Withdrawal of Tentative

Tentative Definition of the Term Aggregate (**C 58 – 28 T**) (accepted Sept. 10, 1963).

Withdrawal of Standard

Standard Definition of the Term Sand (**C 58 – 28**) (accepted Sept. 10, 1963).

This report has been submitted to letter ballot of the committee, which consists of 9 voting members; 7 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

F. W. REINHART,
Chairman.

R. P. LUKENS,
Secretary.

REPORT OF COMMITTEE E-9
ON
FATIGUE

Committee E-9 on Fatigue held two meetings during the year: on June 26, 1962, in New York, N. Y., and on Feb. 5, 1963, in Montreal, Canada. Technical sessions were held at the New York meeting. The Third Symposium on Fatigue of Aircraft Structures was sponsored at the Fourth Pacific Area National Meeting in Los Angeles, Calif. in October, 1962. The Executive Subcommittee also held a meeting in Los Angeles on Oct. 3, 1962.

During the year seven new members were elected to the committee. Total membership is now 84, including consultants and correspondents.

The traditional practice of devoting the latter portion of each Committee E-9 main meeting to an informal presentation of a timely fatigue topic was continued. At the June meeting A. K. Head, Visiting Professor of Engineering, Brown University, discussed "Crack Propagation in Fatigue." At the February meeting, J. DuBuc, Ecole Polytechnic, Montreal, discussed "Low-Cycle Fatigue."

During the year the first 5-yr Bibliography on Fatigue (*STP 9-AA*) covering the period of 1950–1954 was published. In addition, the February, 1963, issue of *Materials Research & Standards* was devoted to three papers on fatigue: "The Mechanism of Fatigue" by G. R. Gohn, "Crack Propagation and Final Failure" by H. F. Hardrath, and "Engineering and Design Aspects" (the 1962 Marburg Lecture) by R. E. Peterson.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1962 Annual Meeting, Committee E-9 presented to the Society through the Administrative Committee on Standards new Tentative Definitions Relating to Fatigue Testing and the Statistical Analysis of Fatigue Data. The definitions were accepted by the Standards Committee on October 31, 1962, and appear in the 1962 Supplement to *Book of ASTM Standards*, Part 3, bearing the designation E 206 – 62 T.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Research (G. M. Sinclair, chairman).—In addition to general discussions of a wide variety of research subjects in the field of fatigue, the subcommittee arranged for speakers for the informal talks following each Committee E-9 meeting. A possible symposium to be held in conjunction with the 1964 Annual Meeting is also being discussed.

Subcommittee II on Papers (P. R. Toolin, chairman) reviewed papers for the three sessions held during the Pacific Area National Meeting in October, 1962, and for two sessions at the Annual Meeting in June, 1963.

Subcommittee III on Survey (W. S. Hyler, chairman).—References on Fatigue for 1960 were collected and published as *STP 9-L*. The first of a series of 5-year bibliographies (for 1950–1954) was published as *STP 9-AA*.

Subcommittee IV on Apparatus and Test Methods (H. R. Neifert, chairman.)—Studies directed toward the formulation of a standard for preparation of R. R. Moore specimens were undertaken. The task of preparing certain sections of the proposed revision of the Manual on Fatigue (*STP 91*) was also undertaken.

Subcommittee V on Aircraft Structures (H. F. Hardrath, chairman) arranged the Third Symposium on Fatigue of Aircraft Structures. Sessions on low-cycle fatigue, helicopter problems, and full-scale testing were held. A panel discussion on low-cycle fatigue was also held. The subcommittee is considering the development of a standard structural fatigue specimen. It has also discussed means of developing closer association with such international groups as the Advisory Group on Aircraft Research and Development (AGARD).

Subcommittee VI on Statistical Aspects of Fatigue (G. R. Gohn, chairman).—Much effort of the subcommittee has gone into the writing of Definitions E 206, which was accepted by the Standards Committee. It is recommended that Definitions E 206 be submitted to the American Standards Assn. for approval as American Standard. The definition of fatigue is being reviewed to insure that it reflects all phases of current knowledge. Because of obsolescence, the Guide for Fatigue Testing and Statistical Analysis of Fatigue Data (*STP 91-A*) has been revised and will be published in the near future.

Task Force on Revision of the Manual on Fatigue Testing (*STP 91*) (F. B. Stulen, chairman).—A complete rewriting of this manual is being undertaken. A large number of topics not included in the original version will be covered. An outline of the manual has been prepared and assignments of responsibility for the various sections have been made. It is expected that two or three years will be required to complete the manual.

This report has been submitted to letter ballot of the committee, which consists of 68 members; 51 members returned their ballots, of whom 50 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

H. J. GROVER,
Chairman.

R. F. BRODRICK,
Secretary.

REPORT OF COMMITTEE E-10
ON
RADIOISOTOPES AND RADIATION EFFECTS*

Committee E-10 on Radioisotopes and Radiation Effects held two meetings during the year including subcommittee meetings: on Oct. 4, 1962, in Los Angeles, Calif., and on Feb. 6, 1963, in Montreal, Canada. Two additional subcommittee meetings were also held at other times during the year.

The committee now consists of 157 members, plus 24 persons who are members of subcommittees but are not members of Committee E-10.

The 1962 Symposium on Radiation Effects on Metals and Neutron Dosimetry[1] drew a large audience at the Pacific Area National Meeting in Los Angeles. Papers from the United States and abroad were of excellent quality. The organization of the entire program was a job well done by Technical Co-chairmen R. Hawthorne and L. E. Steele. Another Symposium on Radiation Effects will be held at the 1963 Annual Meeting. Plans are also being made for a 1964 Symposium on Fast Particle Effects.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee E-10 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[2] All of the existing tentatives referred to in this report appear in their present form in the 1961 Book of ASTM Standards, Part 3, or in the 1962 Supplement.

New Tentatives

as appended hereto:[3]

Tentative Methods for:

Determination of Uranium by Controlled-Potential Coulometry,

Radiochemical Determination of Cesium-137 in Aqueous Solution (Chloroplatinate Method), and

Atom Per Cent Fission in Uranium Fuel (Radiochemical Method.)

Adoption of Tentatives as Standard Without Revision

Tentative Recommended Practice for:

Measuring Neutron Flux Environment for Reactor Irradiated Specimens (**E 199 - 62 T**).

Tentative Definitions of Terms Relating to:

Dosimetry (**E 170 - 60 T**).

Tentatives Continued Without Revision

Tentative Method for Radiochemical Determination of Cesium-137 in Aqueous Solutions (**E 198 - 62 T**).

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] Published as *ASTM STP No. 341*.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[3] The new tentatives appear in the 1963 Supplement to *Book of ASTM Standards*, Part 3.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Education and Information (Richard R. Quinn, chairman).—Due to the pressure of business, Gordon R. Molesworth resigned as chairman of this subcommittee which he helped start. The subcommittee is about ready to release a speaker's list, and printed information on Committee E-10. Plans are being formulated for further service to other committees.

Subcommittee II on Radiation Induced Changes in Materials (J. V. Alger, chairman) has added a fifth task group that will cover space radiation. To serve the pressing national need for information in its area of interest, this subcommittee has sponsored a symposium in 1962,[1] has another nearing completion for 1963, with still a third in process for 1964. Several tentatives are in process with two revisions of existing standards being worked on.

Subcommittee III on Tracer Applications (W. S. Lyon, chairman) is currently reviewing past tentatives, and will have a proposed tentative ready for the Annual Meeting. Four future tentatives are in process and several counting methods are in the discussion stage. Liaison is being maintained with Subcommittee V in this respect. Preliminary symposium plans are under consideration.

Subcommittee IV on Measurements Using External Sources (John L. Kuranz, chairman) has two tentatives, one on moisture and another on density determination, nearing final form. A survey of the needs of industry for standards is about ready for release. A joint symposium with Subcommittee III is under consideration.

Subcommittee V on Dosimetry (D. N. Sunderman, chairman).—J. B. Trice resigned as chairman of this subcommittee. He was very active in its work and greatly responsible for its growth and effectiveness. The Subcommittee has ten tentatives in various stages of completion, and has recommended two tentatives for adoption as standard and a new tentative as noted earlier in this report. A new task group of 32 members on *n*-Dosimetry has been formed making a total of three task groups for this subcommittee which now totals 160 members and observers.

This report has been submitted to letter ballot of the committee, which consists of 108 members; 68 members returned their ballots, of whom 51 have voted affirmatively, and 0 negatively.

Respectfully submitted on behalf of the committee,

G. W. POMEROY,
Chairman.

R. R. QUINN,
Acting Secretary.

REPORT OF COMMITTEE E-11
ON
QUALITY CONTROL OF MATERIALS

Committee E-11 on Quality Control of Materials held two meetings during the year: in New York, N. Y., on June 26, 1962, at the time of the Annual Meeting; and on Nov. 14, 1962, at Society Headquarters in Philadelphia. The Advisory Committee met on the days just prior to the meetings of the full committee.

Two symposia were sponsored by Committee E-11 during the year. A Symposium on Applied Statistics in Materials Testing was held on Oct. 3, 1962, in Los Angeles, Calif., during the Fourth Pacific Area National Meeting. A paper was presented by Grant Wernimont of the Eastman Kodak Company and John Mandel of the National Bureau of Standards explaining the proposed manual on interlaboratory test methods; and another paper was presented by R. B. Murphy of the Bell Telephone Laboratories explaining the reasoning for the recommended practice on definitions of the terms precision and accuracy (E 177). Professor E. P. Coleman of the University of California gave a paper on reliability testing; and Allen Chop of Lockheed Missiles and Space Company gave a paper on Polaris testing and data analysis.

A Symposium on the Application of Statistics to Testing was sponsored by Committee E-11 during Committee Week in Montreal on Feb. 4 to 7, 1963. C. de Courval of Canadian Industries, Ltd. presented a paper on the statistical evaluation of water pollution; G. L. Cockhill of Shawinigan Chemicals, Ltd. presented a paper on an application of response surface principles; and P. F. Wade of Price Waterhouse and Company presented a paper on test methods, specifications, and costs.

During the year Committee E-11's manual for evaluating test methods by a cooperative study among several laboratories was published as *ASTM Manual for Conducting an Interlaboratory Study of a Test Method, ASTM STP 335*. The manual represents the culmination of many years of effort on the part of the committee.

At its November, 1962, meeting, Committee E-11 voted to reorganize its structure so that it might better serve other technical committees of the Society. A special subcommittee was appointed to revise the regulations governing E-11 to bring them into accord with the approved changes in organization.

RECOMMENDATIONS AT ANNUAL MEETING

Tentatives Continued Without Revision

Tentative Recommended Practices for:

Designating Significant Places in Specified Limiting Values (**E 29 – 60 T**),

Use of the Terms Precision and Accuracy as Applied to Measurement of a Property of a Material (**E 177 – 61 T**), and

Dealing with Outlying Observations (**E 178 – 61 T**).

Respectfully submitted on behalf of the committee,

C. A. BICKING,
Chairman.

A. J. DUNCAN,
Secretary.

REPORT OF COMMITTEE E-12

ON

APPEARANCE OF MATERIALS*

Committee E-12 on Appearance of Materials held one meeting during the year: on June 25 and 26, 1962, in New York, N. Y., during the Annual Meeting of the Society. The Advisory Subcommittee and several task groups met in New York, N. Y., on March 12, 1962.

During the year, activities have continued across an increasing spectrum of interests. A permanent section on Methods Review (B. W. Preston, chairman) has been organized under Subcommittee I on Intercommittee Relations.

An important matter now being considered is the proposal of the American Standards Assn. that ASTM assume the sponsorship of Sectional Committee Z58 on Standards for Optics. A formidable task is involved in updating existing American Standards in this field.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee E-12 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] The existing tentatives referred to appear in their present form in the 1961 Book of ASTM Standards, Parts 6, 8, and 9.

New Tentative

Tentative Definitions of Terms Relating to Carbon Paper and Inked Ribbon Products and Images Made Therefrom as appended hereto.[2]

Adoption of Tentatives as Standard Without Revision

Tentative Recommended Practices for:

Goniophotometry of Transmitting Objects and Materials (**E 166 - 60 T**), and

Goniophotometry of Reflecting Objects and Materials (**E 167 - 60 T**).

ACTIVITIES OF SUBCOMMITTEES AND TASK GROUPS

Subcommittee I on Intercommittee Relations (R. J. Fabian, chairman) has organized a new section on Methods Review. The methods concerned, at least in part with some measure of appearance, that are being studied on request for possible improvement include: (1) Color of Clear Liquids (Platinum-Cobalt Scale) (D 1209), (2) Testing Cellulose Acetate Propionate and Cellulose Acetate Butyrate (D 817),

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[2] The new tentative appears in the 1963 Supplement to *Book of ASTM Standards*, Part 6.

(3) Clarity and Cleanness of Paint Liquids (D 2090), (4) Transparency of Plastic Sheeting (D 1746), and (5) Turbidity of Industrial Water (D 1889).

Subcommittee III on Pictorial Representation (J. M. Hemphill, chairman).—A proposal, now ready for interlaboratory testing, should lead to a recommended practice for industrial photography.

Task Group 1 on Manual on Appearance (I. Nimeroff, chairman).—The body of relevant information has been expanded by papers presented by individual members of this group which will be incorporated in the manual.

Task Group 3 on Goniophotometric Methods (I. Nimeroff, chairman) has recommended adoption as standard of Recommended Practices E 166 and E 167 as noted earlier in this report.

Task Group 5 on Typewriter Ribbon and Carbon Paper (W. E. Grady, chairman) prepared the definitions mentioned earlier in this report. It has under active study and development eleven methods of tests for typewriter ribbon and carbon paper.

Task Group 9 on Combining and Updating ASTM Methods D 307 and D 791 (Paul Giesecke, chairman). — These methods are basic to colorimetry. This project was proposed by Committee D-20 on Plastics and will also meet needs expressed by the International Organization for Standardization, through its Technical Committee 61 on Plastics. An international task force, in the latter's working Group 5 on Physical and Chemical Properties, is attempting to reconcile divergent views on this subject, looking toward producing an ISO recommendation. The method being developed by Committee E-12 will undoubtedly provide the basis for the ultimate ISO draft proposal.

This report has been submitted to letter ballot of the committee, which consists of 60 members; 43 members returned ballots, of whom 37 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

GEORGE W. INGLE,
Chairman.

PAUL GIESECKE,
Secretary.

REPORT OF COMMITTEE E-13
ON
ABSORPTION SPECTROSCOPY*

Committee E-13 on Absorption Spectroscopy met twice during the year: in New York, N. Y. on Nov. 15, 1962, in conjunction with the Eastern Analytical Symposium, and on March 4, 1963, with the Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy. The committee plans to meet at the same conventions in November, 1963, and March, 1964.

In addition to the meeting of the committee at the Eastern Analytical Symposium, E. J. Rosenbaum, chairman of Committee E-13, presided at sessions on "Evaluation of Spectrophotometric Performance" that included five papers[1] by experts in the field and a panel discussion.

At the Pittsburgh Conference, E. C. Dunlop, Committee E-13 member, presided at a Symposium on Storage and Retrieval of Analytical Data arranged on behalf of the Society by Committee E-13 with the cooperation of the Society for Applied Spectroscopy.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee E-13 requests approval by the Society at the Annual Meeting of the following recommendations recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[2] The existing tentatives referred to appear in their present form in the 1961 Book of ASTM Standards, Part 7, or the 1962 Supplement.

Proposed Method to be Published as Information

Proposed Recommended Practices for Describing and Measuring the Performance of Spectrophotometers, as appended hereto.[3]

Revision of Tentative

Tentative Definitions of Terms and Symbols Relating to Absorption Spectroscopy (**E 131 – 62 T**):

Change or add the following definitions in their proper alphabetical sequence:

Angstrom, A.—A unit of length equal to 10^{-10} m.

Spectrometer.—An instrument with an entrance slit and one or more exit slits, with which measurements are made either by scanning the spectral range, point by point, or by simultaneous measurements at several spectral positions. The quantity measured is a function of radiant power.

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] Copies of the papers are not available except perhaps by correspondence with the individual authors. The authors' names and abstracts of the papers are given in the official program of the symposium.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[3] The Proposed Method appears in the *1964 Book of ASTM Standards*, Part 31.

Analytical Curve.—The graphical representation of a relation between some function of radiant power and the concentration or mass of the substance emitting or absorbing it.

Adoption of Tentative as Standard With Revision

Tentative Recommended Practices for General Techniques of Ultraviolet Quantitative Analysis (**E 169 – 60 T**):

Section 8(b).—Add a new sentence at the end to read "If dilution is made, C_s is not the concentration in the cell at the time the absorbance is determined; the concentration in the cell is C_s/f."

Section 8(c).—Change to read as follows:

Standard Samples.—The absorptivity of the absorbing material, the concentration of which it is desired to determine, is obtained by examination of a pure sample of this material which is called a standard sample. However, if no such pure material is available, the best available sample is used, or a value of the absorptivity is taken from the literature. Care should be taken to specify this, by reporting values as "percentage against standard sample" or by noting that the accuracy of the analysis is dependent upon a published value of the absorptivity or molar absorptivity. (A reference must be cited.)

Tentative Continued Without Revision

Tentative Recommended Practices for General Techniques of Infrared Quantitative Analysis (**E 168 – 60 T**).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Apparatus Specifications (K. B. Whetsel, chairman) prepared the Proposed Recommended Practices for Describing and Measuring the Performance of Spectrophotometers, referred to earlier in the report. A more rigorous method of evaluation is the main subject of current deliberations.

Subcommittee II on Methods (R. T. O'Connor, chairman) intensively reviewed the Recommended Practices E 168 and E 169, referred to earlier in the report. As a result of this review, E 168 will be revised extensively and E 169 is being recommended for adoption as standard.

The Recommended Practices E 204, issued as tentative in 1962, are based upon conventional techniques of spectrophotometry. Recommended practices for microtechniques are being assembled.

Subcommittee III on Standard Data (W. C. Kenyon, chairman) published a Special Technical Publication entitled "Molecular Formula List of Compounds, Names, and References to Published Infrared Spectra."[4] This publication, prepared mainly through the efforts of L. E. Kuentzel, vice-chairman of Committee E 13, lists 43,500 compounds whose infrared spectra have been coded to date by the subcommittee into the Wyandotte-ASTM (Kuentzel) Punched-Card Index of Absorption Spectral Data.[5] The cards are punched with a unique code to the literature reference. To facilitate translation of the code and to encourage universal adoption of the system of literature-reference abbreviation ("Coden"), a publication will be issued shortly to present this information in convenient tabular forms. Methods of handling all information coded in the cards by simplified visual indexes and by magnetic-tape technology are being developed.

Subcommittee IV on Nomenclature (W. P. Tyler, chairman) prepared the revision of Definitions E 131 given earlier in the report. Other definitions related to the terms and symbols in the recommended practices have priority in future deliberations.

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[4] *ASTM STP 331* is available from ASTM Headquarters.

[5] The Card Index may be purchased from ASTM Headquarters.

This report has been submitted to letter ballot of the committee, which consists of 98 members; 81 members returned their ballots, of whom 57 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

E. J. ROSENBAUM,
Chairman.

R. F. ROBEY,
Secretary.

REPORT OF COMMITTEE E-15
ON
ANALYSIS AND TESTING OF INDUSTRIAL CHEMICALS*

Committee E-15 on Analysis and Testing of Industrial Chemicals held two meetings during the year: on June 28 and 29, 1962, in New York, N. Y., and on Feb. 21 and 22, 1963, in Philadelphia, Pa.

The committee consists of 148 individuals representing 82 companies and organizations, 19 representatives of other committees of the Society, and 5 consulting members.

At the February, 1963, meeting, and subsequent to the amendment of the by-laws to include a third vice-chairman, the following appointments were made: E. N. Luce, as Vice-Chairman, and J. Mitchell, Jr., as Member-at-Large of the Executive Committee.

The following subcommittee chairmen were appointed:

Subcommittee 1 on Editorial and Nomenclature, W. W. Becker.

Subcommittee 2 on Functional Groups, S. Siggia.

Subcommittee B-4 on Organic Acids and Derivatives, E. Willan Wilson.

Subcommittee B-5 on Alumina, J. R. Churchill.

The USA National Committee for ISO/TC 47 on Chemistry sponsored by Committee E-15 met twice during the year under the chairmanship of G. T. McBride, Jr. Because of information on international activity on analyses for alumina received through this subcommittee, a representative of the American alumina industry attended the meeting of ISO/TC 47's Working Group 8 in Italy, November, 1962. A report indicating the desirability of greater American participation in this area was presented to the subcommittee by J. R. Churchill. Eight new members representing new areas of industry and additional technical associations and committees were added to USA National Committee ISO/TC 47 on Chemistry.

A special report on activities of Committee E-15 has been issued to technical journals. The report emphasizes the new areas of activity proposed by a special Advisory Panel.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee E-15 requests approval by the Society at the Annual Meeting of the following recommendations. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] The existing tentative referred to in this report appears in its present form in the 1962 Supplement to Book of ASTM Standards, Part 8.

New Tentatives

As appended hereto:[2]

Proposed Tentative Methods for:

Hydroxyl Groups by Acetic Anhydride Acetylation,

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[2] The new tentatives appear in the *1964 Book of ASTM Standards,* Part 31.

Analysis of Sulfuric Acid, and
Analysis of Hydrochloric Acid.

Revision of Tentative

Tentative Recommended Practice for Developing Precision Data on ASTM Methods for Analysis and Testing of Industrial Chemicals (E 180 - 62 T):

Revise as appended hereto.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee 2 on Standards and Reagents (S. M. Tuthill, chairman) has completed interlaboratory precision studies on standardization procedures for silver nitrate and ammonium thiocyanate solutions. On the basis of results obtained, the procedures were modified, and a second study is under way. Interlaboratory studies on procedures for standardization of potassium dichromate and potassium permanganate have been organized.

Subcommittee 3 on Precision and Accuracy (R. J. Sobatzki, chairman).—The revised Recommended Practice E 180 is proving a valuable guide to other subcommittees of Committee E-15. A further revision of this tentative, as appended to this report,[3] provides clarification but no changes in statistical approach. Now that the subcommittee has established a procedural basis for evaluation of methods, it proposes to concentrate on aiding the interlaboratory studies of other subcommittees, both in the determination of significant variables and in the statistical evaluation of data obtained in methods evaluation.

Subcommittee 4 on Sampling (A. Bloomberg, chairman).—Abstracts have been made of pertinent articles selected from the literature search on sampling sponsored by the Monsanto Chemical Co. at Battelle Memorial Inst. under former chairman H. O. Hehner. The abstracts and the original papers have been distributed to the Task Group on Liquid Sampling (H. Behrend, leader) and the Task Group on Solid Sampling (H. C. Hosford, leader). These task groups are preparing preliminary drafts on recommended general practices for sampling simple solid and liquid materials.

Subcommittee A-1 on Elements (L. Gard, chairman) has had three active task groups:

(1) The Task Group on Chlorine and Sulfur (A. P. Scanzillo, leader) has conducted a second interlaboratory investigation on the significant variables in the Parr bomb sodium peroxide fusion method using the experimental design of Recommended Practice E 180. Based on the information obtained, a further study of the method is being made within the task group prior to a broad interlaboratory evaluation.

(2) The Task Group on Phosphorus and Silicon (D. K. Banerjee, leader) completed the investigation of a third draft of a Parr bomb sodium peroxide fusion method for determining phosphorus and silicon in organic compounds. After presentation of the data, it was concluded that further work on critical factors was necessary. Sub-groups on phosphorus and silicon will determine these factors and how to control them.

(3) The Task Group on Schöniger Combustion (W. Clavan, leader) has a tentative draft of a procedure for decomposing organic materials by combustion in an oxygen-filled flask. Data from this procedure will be compared to that obtained by the Task Group on Sulfur using the Parr bomb fusion method.

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[3] The revised tentative appears in the *1964 Book of ASTM Standards*, Part 31.

Three new task groups were activated: Iron in Trace Amounts (G. B. Wengert, leader), Chloride in Trace Amounts (S. T. Hirozawa, leader), and Sulfur in Trace Amounts (J. W. Alexander, leader). The initial activity in all three is review of existing methods, including those in ASTM publications.

The issuance of a general standard with an "E" designation for determining nitrogen by Kjeldahl digestion is being considered. C. O. Willits is reviewing the feasibility of drafting such a method directly from the Association of Official Agricultural Chemists' procedure and existing ASTM "D" standards.

Subcommittee A-2 on Functional Groups (S. Siggia, chairman) has three active task groups:

(1) The Task Group on Hydroxyl (T. P. Callan, leader) submitted as a new tentative its direct reflux acetylation method, the Tentative Method of Test for Hydroxyl Groups by Acetic Anhydride Acetylation, as appended to this report.[2] This method had been published as information in the 1962 Annual Report. Revised drafts of methods for determining hydroxyl groups by pressure bottle acetylation and by acetylation with perchloric acid catalysis have been prepared; they will be evaluated on four representative compounds in seven participating laboratories.

(2) The Task Group on Unsaturation (E. N. Luce, leader) has been studying two methods for determining unsaturation of aliphatic organic chemicals. These methods are the pyridine sulfate dibromide (PSDB) method and coulometric titration. The Proposed Method of Test for Total Bromine Number of Unsaturated Aliphatic Chemicals, based on the PSDB procedure was published as information in the 1962 Annual Report. The group now has agreed to concentrate on the coulometric method. Coulometers have been evaluated and basic design found to be adequate. Evaluation and development are continuing.

(3) The Task Group on Organic Peroxides (D. K. Banerjee, leader) continued its survey of methods for assaying peroxides. Of a number of methods reviewed, the following were selected for further study: the coulometric titanium procedure of Probiner, the spectrophotometric iodometric method of Heaton and Uri, the diphenyl-*p*-phenylenediamine method of A. Ryland, and a coulometric titration procedure. Work to date indicates the unlikelihood of a single general procedure applicable to all types of peroxides.

Information is being collected on sampling of peroxides, their formation in solvents, methods for determining low concentrations, and on hazards and safe practices in handling peroxides.

Subcommittee A-3 on Physical Properties (E. N. Luce, acting chairman) has three active task groups:

(1) The Task Group on Melting and Freezing Points (J. L. Mottern, leader) cooperated with Subcommittee B (R. F. Shertzer, chairman) of Committee D-16 on the revision of Method D 1493. The revision, providing a general standard for determining solidification point of industrial organic chemicals, will be submitted to the full Committee D-16 for approval.

An initial interlaboratory evaluation of a method for determining melting range by capillary tube showed quite poor reproducibility between laboratories. The method was redrafted and is now being restudied in a second interlaboratory program.

(2) The Task Group on Dissolution Temperature (B. F. Branting, leader) circulated four industrial procedures for determining dissolution temperature. The methods were designed for application to ammonium nitrate, urea, and

ammonia solutions but are capable of more general use. Other procedures are to be reviewed as well, and a standard of broad applicability to industrial chemicals will be developed.

(3) The Task Group on Density-Volume Relationships for Industrial Liquid Chemicals (B. H. Cummings, leader).—The tables provided in Method E 201 have aroused considerable interest and there have been a number of requests to have the information extended to other liquid chemicals, which are being considered.

Subcommittee B-1 on Mineral Acids (W. Clavan, chairman) has four task groups:

(1) The Task Group on Sulfuric Acid (N. C. Schultze, leader) prepared the New Tentative Method for Analysis of Sulfuric Acid, as appended to this report,[2] which describes methods for determining total acidity, Baumé gravity, nonvolatiles, reducing substances, and iron. Twelve laboratories cooperated in evaluating these methods.

(2) The Task Group on Hydrochloric Acid (R. E. Richard, leader) prepared the Tentative Method for Analysis of Hydrochloric Acid, as appended to this report,[2] which describes methods for acidity, Baumé gravity, sulfated ash, sulfur, color, and iron.

(3) The Task Group on Hydrofluoric Acid (B. W. Carr, leader) has been engaged in drafting methods for determining acidity, sulfuric acid, sulfur dioxide, iron, and fluosilicic acid in various grades of hydrofluoric acid. In addition, a method will be submitted for determining water in anhydrous hydrofluoric acid.

(4) The Task Group on Phosphoric Acid (R. M. Ludwig, leader) has reviewed the requirements on this product and concluded that the two industrial types of phosphoric acid—furnace acid and wet process acid—are sufficiently different to be considered as separate products. A questionnaire will be sent to consumers and to suppliers to determine which analytical methods are needed for each of the types.

Subcommittee B-2 on Alkalies (R. J. Funston, chairman) has completed plans for an interlaboratory study of the following tests on sodium hydroxide: total alkalinity as sodium oxide, sodium sulfate, sodium chloride, and iron. Samples of potassium hydroxide will be included in the study as it is expected that methods satisfactory for sodium hydroxide will be satisfactory for the potassium analogue also.

The Task Group on Sodium Carbonate in Sodium Hydroxide (C. Schmiege, leader) conducted an interlaboratory evaluation of gravimetric and volumetric methods for this determination. The data obtained are being examined statistically.

A Task Group on Sodium Carbonate and Sodium Bicarbonate (G. Oplinger, leader) was established to organize the program for developing standard methods of analysis for these industrial chemicals.

Subcommittee B-3 on Alcohols (W. R. Deitz, chairman) has two task groups:

(1) The Task Group on Polyalcohols (R. L. Anderson, leader).—Modification of the mercuric chloride-diphenylcarbazone procedure for trace chlorides was investigated; an alternative coulometric procedure was proposed for study. The activity of the group is being extended to diethylene and triethylene glycols; organization of the program has commenced. Gas chromatographic analyses are believed necessary, and the desired levels of precision and accuracy are to be determined by means of a questionnaire to members of the subcommittee.

(2) The Task Group on Methanol (J. L. Carlson, leader) reviewed and evaluated first drafts of methods for

determining chlorides, acetone, hydrocarbons, carbonizable substance, iron, and water in methanol. Four methods are ready for second drafts; studies are not completed on the others. Data from gas chromatographic analyses were reviewed.

Subcommittee B-4 on Organic Acids and Derivatives (E. Willan Wilson, chairman) was organized at the February, 1963, meeting. Two task groups, one on monobasic acids and one on dibasic acids, have been proposed. A poll of the membership of Committee E-15 will be conducted to determine areas of specific interest.

Subcommittee B-5 on Alumina (J. R. Churchill, chairman) was established at the February, 1963, meeting after a report by J. R. Churchill on the recent meeting of Working Group 8, ISO/TC 47 on Chemistry, indicated the need for American activity on this industrial chemical. Consumers and suppliers of alumina were invited by ASTM to participate and to send representatives to the organization meeting of the subcommittee at Pittsburgh, on March 6, 1963.

Work Group on n-Butyllithium (A. B. Chandler, leader) continued evaluation of several methods for the assay of *n*-butyllithium.

This report has been submitted to letter ballot of the committee, which consists of 144 individuals; 89 returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

E. G. WIEST,
Chairman.

R. C. JOHNSON,
Secretary.

REPORT OF COMMITTEE E-17
ON
SKID RESISTANCE*

Committee E-17 on Skid Resistance held two meetings during the year: in New York, N. Y., in June, 1962, and in Knoxville, Tenn., on Nov. 6, 1962.

During the previous year the committee served as the principal agency involved in the planning of the 1962 Skid Test Correlation Study, sponsored by the Virginia Council of Highway Investigation and Research. The study was held in Tappahannock, Va., in August, 1962, with many members of the committee participating. The results of this study have been made available to the subcommittees for analysis and the report has been submitted to the Society for publication.[1]

The Committee E-17 test tire has been used by many organizations throughout the year, and appears to be performing satisfactorily. Preparation of a specification to cover manufacture of this tire is in progress.

This report has been submitted to letter ballot of the committee, which consists of 66 voting members; 49 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

E. A. Whitehurst,
Chairman.

J. H. Dillard,
Secretary.

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] Available as *STP No. 366.*

REPORT OF COMMITTEE E-18
ON
SENSORY EVALUATION OF MATERIALS AND PRODUCTS

Committee E-18 on Sensory Evaluation of Materials and Products met in New York, N. Y., on June 25 and 26, 1962, and on Jan. 17 and 18, 1963, in Washington D. C.

The committee membership as of Feb. 1, 1963, was 71.

Activities of Subcommittees

Subcommittee I on Nomenclature and Definitions (H. G. Schutz, chairman) is preparing a list of 200 defined terms specifically related to the methodology of sensory evaluation.

Subcommittee II on Principles of Psychophysical Test Methods (W. H. Danker, chairman) is preparing an information circular on concentrations of odorous vapors in test chambers. Work is in progress on the examination of mathematical relationships among sensory difference tests, on a bibliography of general methods for sensory evaluations, and on the anatomical and physiological basis for taste.

Subcommittee III on Preparation of Recommended Practices (D. R. Peryam, chairman) is preparing information circulars on (1) evaluation of suprathreshold intensity, (2) number of panel members for discriminative evaluation, (3) preparation and presentation of samples, and (4) selection of odor observers.

Subcommittee IV on Instrumental-Sensory Relationships (R. A. Baker, chairman) is continuing an extensive survey of industrial and other experiences in establishing correlations between sensory evaluations and instrumental information.

This report has been submitted to letter ballot of the committee, which consists of 71 voting members; 58 members returned their ballots, of whom 57 voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

Amos Turk,
Chairman.

Frederick Sullivan,
Secretary.

REPORT OF COMMITTEE E-19
ON
GAS CHROMATOGRAPHY

The first annual meeting on the Practice of Gas Chromatography was held in East Lansing, Mich. on Sept. 26 to 28, 1962, in cooperation with the College of Engineering and the Continuing Education Services of Michigan State University. Meeting sessions devoted to the discussion of various aspects of the techniques of gas chromatography were held along with meetings of the several subcommittees. Instrumentation information facilities were available to inform registrants on the capabilities and specifications of available chromatographic systems. Registrants totaled 304 persons.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Definitions, Nomenclature, and Standard Data (B. O. Ayers, chairman) has organized several task groups to carry out its planned work. The first accomplishment has been the preparation of the *Compilation of Gas Chromatographic Data, ASTM STP 343*, using a system developed by J. S. Lewis of the Tennessee Eastman Co. Editing of the material was undertaken by Task Group 1 of the subcommittee.

Subcommittee II on Programs and Papers (N. Brenner and C. H. Orr, co-chairmen) was responsible for the organization and conduct of the annual meeting on the Practice of Gas Chromatography in East Lansing, Mich. Preparations for a second annual meeting have been completed and scheduled for Sept. 16 to 18, 1963, in Atlantic City, N. J. The third annual meeting will be held Oct. 14 to 16, 1964, in Houston, Tex.

Subcommittee III on Research (M. K. Testerman, chairman) is continuing its plans to bring the latest in techniques and instrumentation to the attention of the committee at the annual meetings. Tentatively, a discussion of gas chromatographic instrumentation in space exploration has been scheduled for the September, 1963, meeting of Subcommittee III.

Subcommittee IV on Coordination and Standardization of Methods (W. C. Jones, Jr., chairman) has several projects in a state of high activity:

Task Group 1 on Standard Format (J. H. Fager, chairman) will present the final revision of the standard format for gas chromatographic methods.

Task Group 2 on Methods Review (R. W. King, chairman) has completed examination of a number of methods promulgated by other ASTM committees and has forwarded recommendations for revision.

Task Group 3 on Method of General Procedures (R. M. Bethea, chairman) has submitted to Subcommittee IV a draft of a method of general gas chromatography procedures. This comprehensive work should be of considerable assistance to the application of gas chromatographic techniques.

At the 1962 annual meeting of Committee E-19, Subcommittee IV presented a statistical analysis of data obtained by a large group of laboratories on a standard sample of hydrocarbons circulated to meeting registrants. This project and report was so well received that a second standard sample, consisting of fatty acids, is being sent for analysis to registrants at the 1963 meeting.

Respectfully submitted on behalf of the committee,

B. F. DUDENBOSTEL, JR.,
Chairman.

C. G. HARRIZ,
Secretary.

REPORT OF COMMITTEE E-20
ON
TEMPERATURE MEASUREMENT*

The formation of Committee E-20 on Temperature Measurement was authorized by the ASTM Board of Directors at its meeting on May 8, 1962, and a steering committee under the chairmanship of R. D. Thompson, Taylor Instrument Cos., was appointed to effect organization. Two subcommittees of Committee E-1 on Methods of Testing, namely 17 on Thermometers, and 32 on Thermocouples for Temperature Measurement formed the basis for the new committee. The work of these subcommittees was transferred to Committee E-20 together with jurisdiction of Specifications for ASTM Thermometers (E 1 - 62), and the Method of Thermal EMF Test of Single Thermoelement Materials by Comparison with a Secondary Standard of Similar EMF Properties (E 207 - 62 T).

The steering committee met on Sept. 19, 1962, and approved a scope which was approved by the ASTM Board of Directors at its meeting Sept. 30, 1962.

An organizational meeting was held on Dec. 13, 1962, in New York, N. Y., with individuals, organizations, and representatives of other societies invited to participate. At this meeting the following officers were elected:

Chairman, R. D. Thompson, Taylor Instrument Cos.

Vice-Chairmen, D. I. Finch, Leeds and Northrup Co.; and J. F. Swindells, National Bureau of Standards.

Secretary, C. D. Starr, Wilbur B. Driver Co.

The following subcommittees were formed:

I. Editorial.
II. Nomenclature.
III. Source Book.
IV. Thermocouples.
V. Liquid-in-Glass Thermometers.
VI. Radiation Pyrometers.
VII. Optical Pyrometers.
VIII. Bi-metallic Thermometers.
IX. Resistance Thermometers.
X. Filled Systems Thermometers.
XI. Miscellaneous Thermometers.

RECOMMENDATIONS FOR SOCIETY ACTION

Committee E-20 requests approval by the Society at the Annual Meeting of the following recommendations, recognizing that all adoptions as standard require confirmation by Society letter ballot. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[1] The existing standard referred to appears in

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*** Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.**

[1] EDITOR'S NOTE.—No action was taken at the Annual Meeting on the Report of Committee E-20 in the absence of a quorum at the session when the report was scheduled for presentation. Alternate procedures were used in securing Society action on the recommendations, set forth herein, as follows:

The Administrative Committee on Standards accepted on Sept. 20, 1963, the new tentative method.

Through the new interim procedure for standards, the Society accepted on Oct. 30, 1963, the revision of Standard Specifications E 1 - 62.

its present form in the 1962 Supplement to the Book of ASTM Standards, Part 8.

New Tentative

Tentative Method for Comparison Calibration of Thermocouples, as appended hereto.[2]

Revision of Standard, Immediate Adoption

(requires nine-tenths affirmative vote)

Standard Specifications for ASTM Thermometers (**E 1 – 62**) (formerly under the jurisdiction of Committee E-1 on Methods of Testing):

Section 2.—Add new Paragraph (*f*) to read as follows, relettering present Paragraph (*f*) as (*g*):

> (*f*) *Contraction Chamber* is an enlargement of the capillary bore which serves to reduce a long length of capillary tubing, or to prevent contraction of the mercury column into the bulb. This chamber will appear below the main scale or between the main scale and an auxiliary scale.

Section 3.—Change Note 3 to read as follows:

> NOTE 3.—Such thermometers manufactured prior to the adoption of the specifications will retain the same official status as those meeting current specifications.

Section 5(a).—Delete the word "borosilicate."

Section 6(b).—Delete the word "borosilicate."

Footnote 4.—Change Footnote 4 to read as follows:

> [4] Corning 8800, Corning 1720, and Jena 2954 III glasses conform to these requirements and have been found satisfactory.

Section 11.—Add the following sentence to this paragraph: "Reference should be made to the Method of Inspection, Test, and Standardization of Etched-Stem Liquid-in-Glass Thermometers (ASTM Designation: E 77)."

Table I.—Make the following changes in Table I:

Partial Immersion Thermometer.—Change the designations of Partial Immersion Thermometers: 1C-58, 2C-58, and 3C-58 to 1C-63, 2C-63, and 3C-63 respectively.

Saybolt Viscosity Thermometer.—For thermometer 19C-62, 19F-62, under the heading Expansion Chamber, change 150 C to 115 C.

Precision Thermometers.—For Precision Thermometers 69C-62, 69F-62 change the special inscription to read: "ASTM 69C or 69F," and add footnote reference *b*. For Precision Thermometer 70C-62, 70F-62 change the special inscription to read: "ASTM 70C or 70F," and add footnote reference *b*.

At the end of this table add the following footnote: "The thermometers shall be marked with the manufacturer's glass name and code number, for example, Corning 1720."

Figure for Thermometer.—Replace this illustration on each page where it appears with a new figure showing the stem ring deleted.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee IV on Thermocouples (D. I. Finch, chairman) met in Montreal during the ASTM Committee Week and formulated plans for carrying out its activities under the jurisdiction of Committee E-20.

Work has been completed on the Method for Comparison Calibration of Thermocouples, and on Standard Temperature-EMF Tables for Thermocouples. A specification for thermocouples, metal sheathed corrosion-resistant for nuclear service is presently being ballotted upon for submission to the Administrative Committee on Standards.

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[2] The new tentative appears in the *1964 Book of ASTM Standards*, Part 30.

Work also includes the following: (1) development of a method of EMF-stability testing of base metal thermocouple alloys, (2) high-temperature performance of all types of ceramic insulating materials, (3) cryogenic performance of thermocouples, (4) method of transient temperature measurement methods with thermocouples, and (5) establishing calibration procedures for platinum 6 per cent rhodium-platinum 30 per cent rhodium thermocouples.

Subcommittee V on Liquid-in-Glass Thermometers (James F. Swindells, chairman).—Work in progress includes the following: (1) development of specifications for a benzene freezing point thermometer for Committee D-16 on Industrial Aromatic Hydrocarbons, (2) development of specifications for a thermometer for the solid point test for aviation fuels for Research Division VII-D of Committee D-2 on Petroleum Products and Lubricants. (3) development of specifications for a wide range thermometer for tar acids distillation for Committee D-16, and (4) development of a revised bulb stability test for high-temperature thermometers.

This report has been submitted to letter ballot of the committee, which consists of 106 voting members; 73 members returned their ballots, of whom 66 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

R. D. THOMPSON,
Chairman.

C. D. STARR,
Secretary.

REPORT OF COMMITTEE E-21
ON
SPACE SIMULATION

The organization meeting of Committee E-21 on Space Simulation was held in Los Angeles, Calif., on April 15, 1963. The scope for the new committee is:

> The development of definitions of terms and nomenclature, methods of test, and specifications for simulated space environment testing of vehicles, components, and materials intended for space applications and missions, and the promotion of knowledge in this field.
>
> The committee will also function as a coordinating agency maintaining liaison with other organizations and ASTM committees working in this field.

The officers elected are as follows:

Chairman, J. C. Simons, Jr.

Vice Chairman, J. D. Pinson.

Secretary, E. N. Borson.

Members-at-Large, G. Deutsch and J. C. Richmond.

Five subcommittees were formed as indicated below.

Activities of Subcommittees

Subcommittee I, Executive Subcommittee (J. C. Simons, Jr., chairman) is defining long-range and immediate objectives toward the development of standards to simulate what is required to give intelligent test results that will show how a vehicle will behave in the actual environment.

Subcommittee II on Nomenclature and Definitions (W. H. Allen, chairman) is surveying existing glossaries in this field to develop practical definitions of terms that will be readily applicable to the standards developed in the committee.

Subcommittee III on Simulation Requirements (D. D. Carlson, chairman) has responsibilities for:

(1) Defining natural and induced environments for planetary and interplanetary space ventures. This will also include the method or procedure used for measuring environments.

(2) Establishing simulation requirements for the various types of tests to be investigated, which will include tests for materials, components, subsystems, and complete vehicle testing.

(3) Evaluating techniques for duplicating or simulating singular and combined environments.

(4) Developing performance specifications or qualification techniques for the various space parameters to be simulated.

Subcommittee IV on Test Methods (R. W. Johnson, chairman) is organizing subgroups to establish uniform standards for testing components and materials that will be rigorous, based upon existing test simulation facilities. Test methods being explored include pressure-density tests, solar and thermal simulation, gravity and magnetic simulation, high energy radiation, meteroroid simulation, man-rating, and acceleration, shock, and vibration.

Subcommittee V on Liaison (B. G. Achhammer, chairman) is developing ties with all formal and informal organizations in this field to ensure efficient use of manpower and test facilities.

Respectfully submitted on behalf of the committee,

J. C. Simons, Jr.
Chairman.

E. N. Borson,
Secretary.

REPORT OF JOINT COMMITTEE

ON

CHEMICAL ANALYSIS BY POWDER DIFFRACTION METHODS[1]

The Joint Committee on Chemical Analysis by Powder Diffraction Methods[1] which is a joint committee of the American Crystallographic Assn. (ACA), the British Institute of Physics (BIP), the National Assn. of Corrosion Engineers (NACE), and ASTM, held a meeting in connection with the ASTM Annual Meeting in New York, N. Y., on June 28, 1962, and the Editorial Board held a meeting in conjunction with the Pittsburgh Diffraction Conference on Nov. 8, 1962, in Pittsburgh, Pa.

Powder Diffraction Data Card Indexes:

The joint committee completed contractual arrangements for the production of a new data retrieval system based on optical coincidence (punched hole) cards to supplement the plain, Keysort, and IBM data cards. Known as the Matthews Index, it gives rapid identification of powder patterns of single phases. With the utilization of negative element cards (to blank out holes of elements known to be missing) mixtures may be identified with facility.

An additional index known as the Fink Index is being prepared in book form. This index lists the eight strongest d-values arranged in eight entries, as against only the three strongest lines arranged as three entries in the Hanawalt index system. This will be a supplement to the other indexes for identification of complex mixtures, and may prove to be better than the Hanawalt index for general purposes.

In preparation is the thirteenth set of data cards, available as plain, IBM, and Keysort cards in the Hanawalt system. This latest set brings the total number of organic and inorganic data cards to 11,000. Included with the thirteenth set and also available separately will be a corrected listing of the alloy phase designations of all alloys now appearing in the powder diffraction data file.

Data Producing Projects:

The original file was established by the joint committee by assembling available powder diffraction data in the public domain. The sale of these organized data permitted the committee to support the development of new patterns as well as to revise or improve existing ones. The following projects are supported by the joint committee:

National Bureau of Standards Associateship (H. F. McMurdie, director).—Established in 1949, associates have been employed who have concentrated on the production of very precise data (noted in the card file by an asterisk or a star).

University College at Cardiff, Wales (A. J. C. Wilson, coordinator) is working on problems of diffractometry and absolute intensity in close liaison with the National Bureau of Standards. This project prepares about 60 high-accuracy powder diffraction patterns per year.

Technisch Physische Dienst, Delft (J. W. Visser, coordinator).—Absolute intensities have been determined for the 17 patterns developed this year. Careful attention has been paid to completeness of data including other than powder data. This group is actively working with The Netherlands mineralogical working committee, part of whose activity is to develop X-ray data. They are also exploring the development of powder patterns for hydrates having a small range of stability.

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[1] This committee was originally (1941) a joint activity between Subcommittee IV on X-ray Methods of ASTM Committee E-4 on Metallography, and National Research Council.

Edsel B. Ford Institute for Medical Research (Jonathan Parsons, coordinator) is preparing X-ray powder diffraction data for 130 steroids. Data on 44 steroids have been submitted for publication. It is hoped to establish a complete list of these important chemicals which control so much of human chemistry.

Birkbeck College Crystallography Laboratory, University of London (J. W. Jeffrey, supervisor) is developing patterns on cement, pulverized fuel, and corrosion products. The degree of accuracy obtainable for quantitative estimations of components in the mixture using a proportional counter diffractometer and discriminator is being investigated.

Five Japanese Universities (Takeo Fujiwara, coordinator) are cooperating to provide new patterns for the file; these are: Hiroshima University (Professor Unegaki), Osaka University (Professor Watanebe), Institute of Solid State Physics (Professor Saito), Tokyo Institute of Technology (Professor Kubo), and Mineral Institute, University of Tokyo (Professor Sadanaga).

British Museum (G. F. Claringbull, supervisor) is developing a large number of mineral patterns.

Mineralogisk Museum, Oslo (H. Neumann, curator) has submitted a large number of mineral patterns, especially of rare minerals found only in Norwegian pegmatites.

Department of Geology, University of Durham (R. Phillips, lecturer) is preparing a complete set of powder patterns for the complex group of amphibole minerals.

University of Michigan (W. C. Bigelow, supervisor) is developing electron diffraction data. Thirteen laboratories have cooperated to develop electron diffraction patterns for about 60 chemicals. These data showed that X-ray and electron diffraction *d*-values represent the same reflections and that most of the high intensity X-ray lines are found in the electron patterns even when highly oriented samples are used. For electron diffraction data, particular attention must be paid to the effects of preferred crystal orientation, rapid fall-off of background intensity, and dynamic scattering effects. Electron diffraction data will be added to the X-ray data file and separate cards added only when the electron lines are significantly different from the X-ray lines.

University of Leeds (M. R. Truter, supervisor) is developing X-ray diffraction data on coordination compounds of transition metals. The entire project is expected to cover some 375 compounds.

Respectfully submitted on behalf of the committee,

W. L. FINK,
Chairman.

J. W. CAUM,
Secretary.

REPORT OF COMMITTEE F-1

ON

MATERIALS FOR ELECTRON TUBES AND SEMICONDUCTOR DEVICES*

Committee F-1 on Materials for Electron Tubes and Semiconductor Devices held three regular meetings during the year: in New York, N.Y., on June 21 and 22, 1962; at Skytop, Pa., on Oct. 31 and Nov. 1, 1962; and in Washington, D.C., Feb. 13 and 14, 1963. In addition, a Symposium on Cleaning and Materials Processing for Electronics and Space Apparatus[1] was sponsored in connection with the Fourth Pacific Area Meeting in Los Angeles in the fall of 1962.

The committee consists of 114 members, of whom 57 are classified as producers, 38 as consumers, and 19 as general interest members.

Speakers of interest during the year were Clarence Luck, Jr., of Raytheon Co., who spoke on "Optical Masers" at the June meeting, and Tom Crumley of General Electric Co., who talked on the subject, "Perspectives in the Apollo Program" at the February, 1963, meeting.

Considerable background was established for handling problems in the microelectronics field, using the present committee organization, as far as possible, to orient the various problems. Principal emphasis was placed on specifications for encapsulants (Subcommittee V), and a start was made with respect to thin films (Subcommittee VI). An editorial check showed approximately 37 related ASTM specifications which may prove of considerable value in the microelectronics field.

The committee has continued its policy of providing both English and metric units in its standards, and has developed a guide for conversion of English to metric units. Liaison was established with national and international organizations. A. Haase and E. Thurber, in particular, rendered excellent liaison service in the effort.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1962 Annual Meeting, Committee F-1 presented to the Society through the Administrative Committee on Standards the following new tentatives which were accepted by the Standards Committee on March 21, 1963:

New Tentative Methods of Test for:

Sizing and Counting Airborne Particulate Contamination in Clean Rooms and other Dust-Controlled Areas Designed for Electronic Work (F 25 – 63 T),

Determining the Orientation of a Semiconductive Single Crystal (F 26 – 63 T),

Preparing a Test Ingot of Germanium by the

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] To be published as *ASTM STP No. 342.*

Hydrogen Reduction of Germanium Dioxide (F 27 - 63 T), and

Measuring the Minority-Carrier Lifetime in Germanium and Silicon (F 28 - 63 T).

RECOMMENDATIONS FOR SOCIETY ACTION

Committee F-1 requests approval by the Society at the Annual Meeting of the following recommendations. The results of the committee ballot on the recommendations will be reported at the Annual Meeting.[2]

New Tentatives

as appended hereto[3]

Tentative Recommended Practices for:

Scratch-Adhesion Testing of Cathode Coatings, and

Determination of Gas Content of Strip Materials.

Tentative Specifications for:

Dumet Wire for Glass-to-Metal Seal Applications,

Iron-Nickel Sealing Alloys, and

42 Per Cent Nickel-6 Per Cent Chromium-Iron Sealing Alloy.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Cathode Materials (C. D. Richard, Jr., chairman) is working in three general areas: (1) electrical tests related to cathode materials and performance, (2) physical and mechanical tests on cathode materials, and (3) fabricated parts and the physical chemistry of the cathode system.

The International Electrotechnical Committee (IEC) in a recent meeting at Nice, France, approved an international standard based on the ASTM Methods of Measuring Interface Impedance Characteristics of Electron Tube Cathodes (F 300), developed by Section A on Electrical Tests. This section is preparing to add new techniques for measuring thin films to the Methods of Test for Sublimation Characteristics of Metallic Materials in Cathode Sleeve Form by Electrical Resistance (F 278), and is continuing to work on cathode temperature measurement.

A method has been developed by Section B on Physical and Mechanical Tests for measuring the diameter of cathode sleeves to very close tolerance (±0.00002 in.) which is to be balloted upon as an addition to the Methods of Testing Sleeves and Tubing for Electron Tube Cathodes (F 128). Work is continuing on collapse strength of cathode sleeves.

Section C on Physical Chemistry prepared the new Tentative Recommended Practice for Scratch-Adhesion Testing of Cathode Coatings appended to this report. Progress is being made toward a nondestructive test for cathode coating adhesion using an interface impedance measurement; a specification for a standard triple carbonate and a standard double carbonate for cathode coating was made ready for ballot of Subcommittee I. A study of binder materials has been initiated.

Subcommittee II on Insulators (M. H. Batlan, chairman).—The Specifications for Aluminum Oxide Powder (F 7), under task group chairman W. H. Davenport, and the Specification for Mica Bridges (F 12) under task group chairman W. C. Downing are being reviewed looking toward the upgrading of these specifications. A review is in progress of new recommended methods and practices for dimensioning mica bridges.

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[2] EDITOR'S NOTE.—No action was taken at the Annual Meeting on the Report of Committee F-1 in the absence of a quorum at the session when the report was scheduled for presentation. Society action on the recommendations, set forth herein, was subsequently taken through the Administrative Committee on Standards. Approval was given to the recommendations by the Standards Committee on Sept. 20, 1963.

[3] The new tentatives appear in the 1963 Supplement to *Book of ASTM Standards*, Part 2.

Subcommittee III on Strip (P. P. Prichett, chairman) prepared the new Recommended Practice for Determination of Gas Content of Strip Materials, appended to this report.

The problem of strip thickness tolerances is not yet resolved. The task group will hold an interim meeting to consolidate all findings and prepare a recommendation for action by Subcommittee III.

A task group is developing a specification for molybdenum and another is investigating methods for measuring surface properties of cathode strip.

Subcommittee IV on Wire (J. R. Howell, chairman).—A task group has sent out questionnaires to interested manufacturers and users of high-purity wire for semiconductor devices to determine interest in developing a specification. The response has been favorable.

A revision of the Method for Bend Testing of Wire for Electronic Devices and Lamps (F 113) has been drafted for subcommittee ballot.

The task group for the Specification for Round Wire for Grid Siderods in Electron Tubes (F 9) will be reactivated.

Additional information will be solicited from industry by the task group of the Specification for Miniature Electron Tube Leads (F 10) to prepare a proposed revision.

Withdrawal of the Specification for Round Nickel Wire for Lamps and Electronic Devices (F 175) has been approved by the subcommittee and a committee letter ballot will be circulated.

An attempt will be made to generate interest on the part of users and manufacturers in development of specifications for lead and pigtail wires for use in micromodules.

The subcommittee approved changes in the Specification for Round Wire for Winding Electron Tube Grid Laterals (F 290) which will now be balloted in the committee.

Subcommittee V on Metallic-Nonmetallic Seals (C. P. Marsden, chairman) is concerned with standard seals, particularly those involving ceramic-metal bonds. Effort has been concentrated on the bar test and thermal shock. With respect to brazing alloys, interlaboratory tests on specimens of copper-silver eutectics for melt tests are in progress. Additional specification work is being done on metallized surfaces with reference to determining thickness and porosity.

Considerable work is in progress on nonmetallic materials for seals, involving recommended practices for flaw detection with dyes and for thermal conductivity. The effort of this group also is directed to determining densities of small ceramic parts, and such qualities as surface roughness. A master specification for alumina is being worked on in conjunction with Committee D-9 on Electrical Insulating Materials.

The group on metal sealing wires completed the new Specification for Dumet Wire for Glass-to-Metal Seal Applications, appended to this report.

Also with further reference to sealing materials, progress was made with respect to the nickel-iron alloys and the nickel - iron - chromium alloy specifications. Specifications F 256 and F 257 on chromium-iron alloys are under revision.

There has been considerable effort on the problems of leak testing, essentially to bring together the knowledge applicable to the leak testing of seals used in the electronics industry.

Subcommittee VI on Semiconductors (H. M. Pollack, chairman).—Of seven proposed new specifications balloted by the committee, three (on orientation, lifetime, and germanium test ingot from germanium dioxide) have been approved by the Administrative Committee on

Standards. The remaining four (on conductivity type, resistivity, Czochralski test crystal, and floating zone test crystal) are being studied further.

The National Bureau of Standards in its work on resistivity measurement of semiconductors found that significant wear had taken place on the probe points after 50 applications of the points to the silicon surface, even under surface conditions much less conducive to wear than the lapped finish on wafers recommended for the actual test.

New task groups were organized to cover materials employed in diffusion of doping elements into semiconductors and photolithographic materials.

A method for oxygen in silicon is undergoing editorial review and is expected to be balloted on by Committee F-1 in June, 1963. Methods for determination of dislocations in silicon and for measurement of resistivity of thermoelectric materials are being developed by the subcommittee.

The newly formed Thin Film Materials task group is studying the materials employed in the evaporation technique for thin film structure formation. Task groups have reported on dislocations in germanium, charge mobility in III-V intermetallic compounds, and epitaxial materials. The method for measurement of epitaxial layer thickness by the infrared interference technique is now being editorially reviewed prior to the Subcommittee VI letter ballot.

Subcommittee VIII, Editorial (E. A. Thurber, chairman) conducted reviews of all new and revised specifications. Each subcommittee provides a liaison member to take care of its interests. This group also effectively contributed toward the use of the metric system in the standards developed by the committee.

Subcommittee IX on Materials Analyses (J. C. Hickle, chairman) is concerned particularly with spectrochemical analyses, serving the other subcommittees. Work during this period included determination of oxygen by inert gas diffusion. A specification was submitted for co-precipitated 4 per cent calcium carbonate and co-precipitated equimolar barium-strontium carbonate used in coating cathode base metals.

With respect to spectrochemical analysis, the group has considered X-ray methods for nickel. Analysis of aluminum oxide, abrasive grade is the subject of an interlaboratory testing program.

Subcommittee X on Control of Contaminants (D. E. Koontz, chairman) sponsored a Symposium on Cleaning and Materials Processing for Electronics and Space Apparatus[1] referred to earlier in this report.

In Section A on Air-Borne Particulate Matter, inspection devices, photometers for measuring air-borne particulate matter, and gas monitoring equipment and methods have been under discussion. Section B on Surface Examination has been concerned with electrical measurements of surface contaminants. The proposed Method for Identification of Particulate Matter by X-Ray Diffraction has been revised and distributed for comment. The procedures for the Weiss ring oven tests will be reworked by R. A. Cotton for further consideration. A particle identification scheme utilizing IBM card methods is being made available to the task group. A revision of the Method of Measuring and Counting Particulate Contamination on Surfaces (F 24) will be requested in order to standardize microscope techniques.

In Section C on Processing Liquids, a water conductivity test and the silting index method for total and suspended solids are being handled in collaboration with Committee D-19 on Industrial Water.

In Section D on Sorbed Gases, the

method on barium flash getters is ready for letter ballot.

This report has been submitted to letter ballot of the committee, which consists of 114 members; 70 members returned their ballots, of whom 62 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

S. A. STANDING
Chairman.

C. L. GUETTEL
Secretary.

REPORT OF COMMITTEE F-2
ON
FLEXIBLE BARRIER MATERIALS*

Committee F-2 on Flexible Barrier Materials held two meetings during the year: on Oct. 4, 1962, in Columbus, Ohio, in conjunction with Committee D-10 on Packaging, and on April 9, 1963, in Philadelphia, Pa.

The committee consists of 84 voting members, of whom 46 are classified as producers, 18 as consumers, and 20 as general interest members.

RECOMMENDATIONS FOR SOCIETY ACTION AT ANNUAL MEETING

Committee F-2 requests approval by the Society at the Annual Meeting of the following recommendations. The results of the committee ballot will be reported at the Annual Meeting.[1] The existing tentative referred to in this report appears in its present form in the 1961 Book of ASTM Standards, Part 6.

New Tentative

Method for Exposing Flexible Barrier Materials to Liquids for Extraction, as appended hereto.[2]

Revision of Tentative

Tentative Definitions of Terms Relating to Flexible Barrier Materials (**F 17 – 61 T**):

Add two new definitions to read as follows:

Seal.—A continuous joint of two or more surfaces of sheet material such as made by fusion or adhesion.

Seam.—A non-continuous joint of two or more surfaces of sheet material such as made by stitching, spot adhesion, or intermittent fusion.

Change the present definitions of "Barrier" and "Flexible" to read as follows:

Barrier.—Any material limiting passage through itself of solids, semisolids, liquids, gases, or forms of energy such as ultraviolet light.

Flexible.—Easily hand-folded, flexed, twisted, and bent.

Delete the present definitions of "Flexible Barrier" and "Permeable."

This report has been submitted to letter ballot of the committee, which consists of 86 members; 37 returned their ballots, of whom 35 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

B. H. MARTIN,
Chairman.

D. L. MCCREADY,
Secretary.

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[2] The new tentative appears in the 1963 Supplement to *Book of ASTM Standards*, Part 6.

REPORT OF COMMITTEE F-3

ON

GASKETS*

Committee F-3 on Gaskets held its organizational meeting in Philadelphia, Pa., on June 6, 1962. Section X on Gaskets of the SAE-ASTM Technical Committee on Automotive Rubber formed the nucleus of the new committee.

The officers elected for the ensuing term of two years are as follows:

Chairman, T. H. Redman.

Vice-Chairmen, F. M. Gavan and G. D'Olier.

Secretary, J. M. Hiscott.

The first regular meeting of the committee was held at Lancaster, Pa. on Oct. 11 and 12, 1962. R. C. Berry was elected secretary to replace J. M. Hiscott who had resigned from the committee, and Agnes Potepan was elected membership secretary. Subcommittees were set up as follows:

I. Administrative, F. M. Gavan, chairman.
II. Methods of Test, J. C. Toman, chairman.
III. Specifications, E. P. Francis, chairman.
IV. Research, R. Sanftner, chairman.

As of Feb. 18, 1963, the committee consists of 60 members, of whom 25 are classified as producers, 23 as consumers, and 12 as general interest members.

The jurisdiction of the following four methods have been transferred to Committee F-3 from Subcommittee VI on Packings, of Committee D-11 on Rubber and Rubber-Like Materials; and the specification listed has been transferred from the SAE-ASTM Technical Committee on Automotive Rubber:

Standard Methods of Testing:

Compressed Asbestos Sheet Packing (**D 733 - 59**).

Tentative Method of Test for:

Compressibility and Recovery of Gasket Materials (**D 1147 - 61 T**),
Sealability of Gasket Materials (**D 2025 - 62 T**), and
Stress Relaxation of a Gasket Material (**D 2139 - 62 T**).

Tentative Specification for:

Nonmetallic Gasket Materials for General Automotive and Aeronautical Purposes (**D 1170 - 62 T**).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Methods of Test (J. C. Toman, chairman) is actively seeking to broaden the conditions of test under which sealability and stress relaxation can be measured. Work is also being conducted or considered on rapid quality control methods and on corrosion, dimensional stability, and compression set.

Subcommittee III on Specifications (E. P. Francis, chairman) is working on an open-ended format for gasket specifications based on functional tests as well as physical tests. Separate sections will be organized for metallics and nonmetallics.

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* Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

Subcommittee IV on Research (R. W. Sanftner, chairman) will be a clearing house for all non-routine gasket requirements and test procedures and will serve as a liaison with other ASTM committees and other pertinent organizations.

This report has been submitted to letter ballot of the committee, which consists of 61 members; 53 members returned their ballots, of whom 52 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

T. H. REDMAN,
Chairman

R. C. BERRY,
Secretary.

REPORT OF COMMITTEE F-4

ON

SURGICAL IMPLANT MATERIALS

Committee F-4 on Surgical Implant Materials was formally organized in Chicago, Ill., on June 14, 1962. The scope of the committee is "the development of definitions of terms and nomenclature, methods of tests, and specifications for materials for surgical implants. This includes basic and composite materials and their applications, performance tests, and the promotion of research in this field."

Five subcommittees have been organized as follows:

Subcommittee I on Compatibility (L. C. Miller, chairman) to determine the effects of implant materials on tissues and the effects of tissues and body fluids on the materials, and to devise test procedures for making such determinations.

Subcommittee II on Mechanical and Physical Properties (H. J. Grover, chairman) to determine the properties of the materials that are significant to the performance of their proper function when implanted in the human body. The properties of bone itself are also to be included.

Subcommittee III on Performance (L. M. Patrick, chairman) to examine the past performance of implants with the object of establishing loads and load histories to which implants are subjected and to devise test procedures.

Subcommittee IV on Specifications (S. E. Tyson, chairman) to write specifications for implant materials based on recommendations of the other subcommittees.

Subcommittee V on Implant Applications (J. K. Wickstrom, chairman) to establish design guides and standards for specific implants such as bone screws, plates, etc., which must be designed to carry loads as part of a composite structure, the other part of which is a living bone.

In order to provide a common background for the committee a symposium covering the interests of the subcommittees was held in Detroit, Mich., on Dec. 14, 1962, and the papers presented have been distributed to the members of the committee. Among the papers was one dealing with the preparation of surgical implant specifications in England.

Since implanted materials include plastics and non-human bone, two chairmen have been appointed to develop symposia covering the use of these materials in surgical implants. It is expected that these symposia will be held from 12 to 18 months in the future.

This committee is probably unique in the history of ASTM in that it numbers more surgeons among its members than are found in all the rest of the ASTM committees combined. For this reason a task force is currently investigating the advisability of preparing a handbook for engineers and surgeons explaining terms and concepts of both fields in a common language. It is expected to have the first tentative specification for metals reported at the next meeting in Philadelphia on Nov. 22 and 23, 1963, and ready for publication by June, 1964.

Respectfully submitted on behalf of the committee,

HERBERT R. LISSNER,
Chairman.

JAMES HITE,
Secretary.

TECHNICAL PAPERS
With Discussions

MECHANICAL PROPERTIES OF COLD-DRAWN MARTENSITIC SAE 4340 STEEL ROD*, †

By E. DiCesare[1]

Synopsis

In the cold drawing of steel rods, the processing conditions can have an appreciable effect on the mechanical properties. Processing variables in this investigation were prior heat treatment, consisting of quenching and tempering at 1150, 1000, 800, or 500 F, or at room temperature; reduction (10 to 40 per cent); back-pull (0 to 16,650 lb); drawing speed (10 to 40 ft per min); and aging temperature (room temperature or 400 F). Final rod diameter was 0.409 in. Engineering tensile, notch tensile, hardness, and true stress - true strain properties are presented. The maximum increase in engineering tensile and yield strengths was realized in the material pretempered at room temperature, drawn 10 per cent, and aged at 400 F. The yield and tensile strengths were increased from 222,000 psi to 368,000 psi and from 316,000 psi to 371,000 psi, respectively, with improved ductility. The primary effect of cold drawing—to strengthen by strain hardening—is dependent upon the pretempering temperature. An additional strengthening increment is due to aging at 400 F and appears to be essentially independent of the pretempering temperatures. Incomplete strain hardening based on the tensile stress-strain curve, the appearance of a sharp yield point or yield point drop for some processing conditions, and the effect of the Bridgman correction on the flow curves were observed and are discussed. Some data are presented to show the effects of reduction, back-pull, and pretempering temperatures on the draw load.

In recent years the combination of deformation with heat treatment has received considerable attention in the search for higher strength metals and alloys. A summary of the numerous combinations that have been employed, together with a bibliography, has been prepared by Radcliffe and Kula (**1**).[2] The combinations have been classed into three principal groups as follows:

Class I—Deformation before austenite transformation,

Class II—Deformation during austenite transformation, and

Class III—Deformation after austenite transformation.

Each group also includes appropriate subclasses based on the transformation product of austenite. The deformation process may be any of those available, such as rolling, stretching, drawing, or forging.

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* Presented at the Sixty-sixth Annual Meeting of the Society.

† The statements and opinions expressed in this article are those of the author and do not necessarily indicate the views or policy of the U. S. Army Materiel Command.

[1] Mechanical Metallurgy Branch, U. S. Army Materials Research Agency, Watertown, Mass.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

The group pertinent to research reported here is Class III, subgroups (*a*) and (*b*) which consist of the deformation of untempered or tempered martensite, respectively, followed by retempering or aging. Several researchers have investigated this area to (1) establish the strengthening mechanism involved and (2) determine the mechanical properties resulting from the treatment (**2–5**).

The SAE 4340 alloy has been of particular interest. The resultant observations and theories have indicated that the primary strengthening mechanism for worked untempered martensite is strain hardening, with subsequent tempering as an additional strengthening increment. For the deformation of tempered martensite followed by aging, there is a further strengthening due to an interaction between the substructure developed during straining and the carbides or carbon, or both, in solution.

A yield point phenomenon is found under some conditions of heat treatment and straining. Stephenson and Cohen (**2**) attribute this to a redistribution of the carbon or carbides and Breyer and Polokowski (**3**) attribute it to a stress-induced ordering mechanism. It is clear that further research is necessary to define the mechanism for the yield point phenomenon.

Increases in tensile strength levels resulting from the thermomechanical treatments have been appreciable. Tensile strengths approaching 400,000 psi have been reported for SAE 4340 steel as-quenched and drawn approximately 9 per cent (**3**); however, the uniform elongation was low and the yield-tensile strength ratio was unity. This is generally true of high-strength martensitic steels processed thermomechanically.

The work reported herein involves the cold drawing of SAE 4340 heat-treated steel rods through a die 0.4096 in. in diameter. Objectives were to determine (1) the smooth and notched tensile properties as a function of (*a*) tempering temperature, (*b*) back-pull, (*c*) per cent reduction, (*d*) drawing speed, and (*e*) aging; and (2) the forces required to draw the rods.

Although relatively few investigations have been conducted into the drawing of rods (**6**), numerous investigations have been conducted on the drawing of wire, including the effect of back tension (**7–9**). Since the drawing of rod and wire are the same basic process, with the exception of material diameter and length, wire drawing research is generally applicable to the rod drawing process. Thus, the general effects of the several variables of the rod drawing process are known. Data obtained in this investigation are presented to show some of these effects.

MATERIALS AND TEST PROCEDURES

The test material consisted of rods from a single heat of cold-rolled SAE 4340 steel, initially 1 in. or $\frac{3}{4}$ in. in outside diameter. These were swaged to various diameters so that reductions of 10, 20, 30, or 40 per cent would be obtained by drawing through a commercial die 0.4096 in. in diameter. All rods were protected from decarburization with No-Carb, and heat treated as follows:

Normalize at 1650 F for 1 hr, air cool
Austenitize at 1550 F for 1 hr, oil quench
Temper 1 hr, air cool:
  Nos. 21 to 36 at 1150 F
  Nos. 1 to 16 at 1000 F
  No. 20 at 800 F
  No. 38 at 500 F
  No. 40 at room temperature

Sufficient material was then cut off each rod for one smooth tension specimen (0.252-in. diameter). The rods were

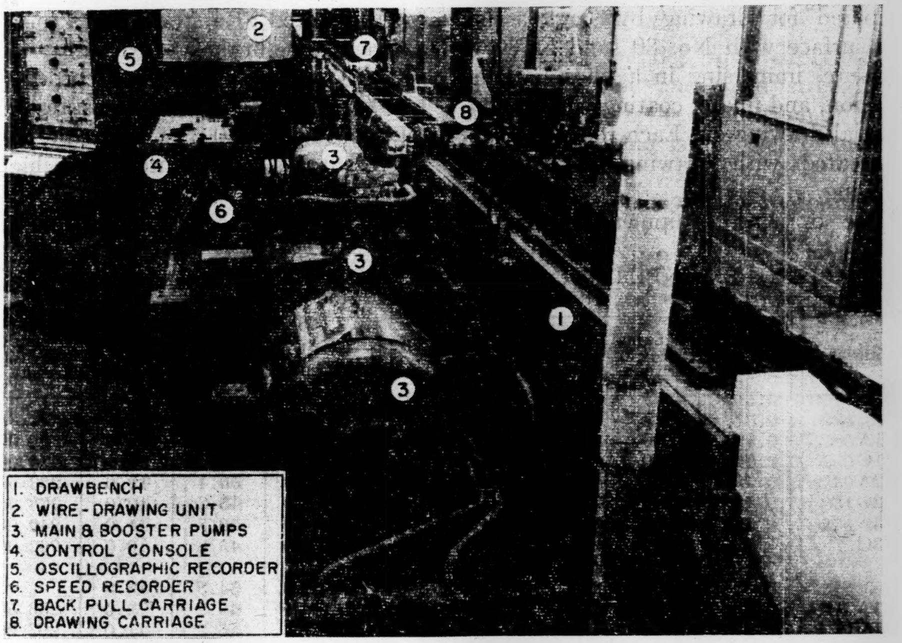
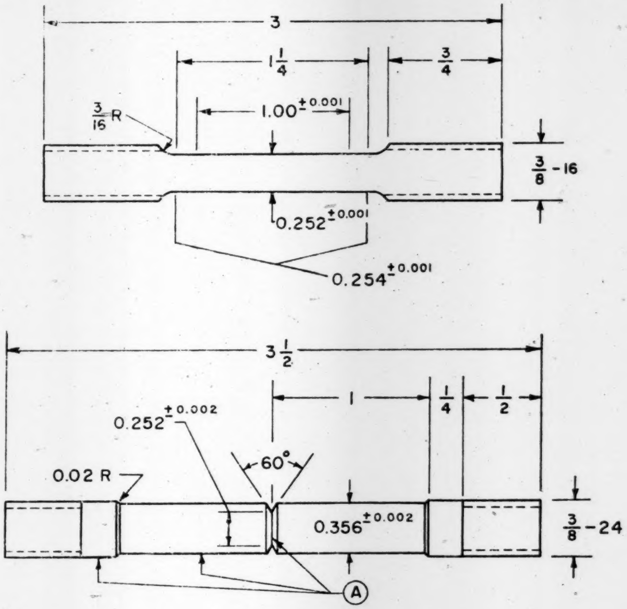

1
2
3
3
3
3
4
5
6
7
8
1. DRAWBENCH
2. WIRE-DRAWING UNIT
3. MAIN & BOOSTER PUMPS
4. CONTROL CONSOLE
5. OSCILLOGRAPHIC RECORDER
6. SPEED RECORDER
7. BACK PULL CARRIAGE
8. DRAWING CARRIAGE

Fig. 1.—Rod- and Wire-Drawing Machine with Instrumentation.


3
1 1/4
3/4
3/16 R
1.00 ±0.001
3/8 - 16
0.252 ±0.001
0.254 ±0.001
3 1/2
1
1/4
1/2
0.252 ±0.002
60°
0.02 R
0.356 ±0.002
3/8 - 24
A

Notes

1. Ⓐ SURFACES TO BE SQUARE AND CONCENTRIC TO WITHIN 0.0005 in.

2. NOTCH RADIUS TO BE 0.001 in. MAX.

Fig. 2.—Tension Test Specimens.

prepared for drawing by sandblasting the surface with No. 80 sand at 80 lb pressure, immersing in a phosphatizing solution, and finally coating with a commercial drawing oil. Each rod was again lubricated with drawing oil prior to drawing.

The drawing equipment, shown in Fig. 1, consists of a rod and a wire drawing machine, each powered by hydraulic equipment and controlled by a servomechanism to within ±1 per cent of the draw load.

TABLE 1.—ENGINEERING MECHANICAL PROPERTIES OF SAE 4340 RODS PRETEMPERED AT 1150 F.

Identification[a]	Reduction, per cent	Drawing Speed, ft per min	Back-Pull, lb	Yield Stress (0.2 per cent), psi	Tensile Stress, psi	Reduction of Area, per cent	Elongation, per cent	Notch Tensile Strength, psi
24	...	...	...	143 800	155 800	53.6	16.0	...
24-1A	10	40	14 850	165 800	168 300	43.4	11.0	252 000
24-1C	10	40	14 850	174 300	177 400	47.4	10.0	270 000
24-2A	10	40	8 450	165 300	170 300	50.4	12.0	253 500
24-2C	10	40	8 450	176 400	180 400	45.8	10.0	268 000
24-3A	10	40	0	162 300	166 300	50.8	11.0	249 000
24-3C	10	40	0	170 800	177 400	45.8	10.0	262 000
28	...	...	...	141 300	154 300	54.8	18.0	...
28-1A	20	25	5 800	165 300	172 800	49.2	12.0	256 500
28-1C	20	25	5 800	173 300	179 900	46.2	11.0	267 000
28-2A	20	25	11 700	166 300	174 300	48.0	11.0	257 000
28-2C	20	25	11 700	180 400	186 400	45.8	11.0	267 000
28-3A	20	25	0	156 300	170 300	50.8	13.0	252 000
28-3C	20	25	0	184 400	188 400	44.0	11.0	262 000
32	...	...	...	140 300	153 300	56.2	18.0	...
32-1A	30	43	5 000	173 800	185 400	47.4	11.0	264 000
32-1C	30	43	5 000	185 400	192 400	44.0	11.0	268 000
32-2A	30	40	8 900	176 400	184 400	45.2	11.0	264 000
32-2C	30	40	8 900	188 400	195 400	42.4	10.0	264 000
32-3A	30	47	0	180 400	187 400	45.2	11.0	260 000
32-3C	30	47	0	186 400	189 900	44.0	11.0	270 000
36	...	...	...	141 300	152 800	35.4	15.0	...
36-1A	40	38	0	184 900	187 900	28.8	7.0	267 500
36-1C	40	38	0	194 400	198 400	24.7	8.0	274 500
36-2A	40	28	4 100	188 400	191 400	24.0	7.0	267 000
36-2C	40	28	4 100	192 400	194 400	33.4	8.0	268 500

[a] A denotes as-drawn; C denotes aged at 400 F for 1 hr after drawing.

Drawing and back-pull loads were measured during the draw by means of load cells consisting of 4 steel blocks approximately 1 by 2 by 8 in. which replaced 2 links of each chain and on which were mounted SR4 strain gages to form a Wheatstone bridge.

An electrical tachometer-recorder system (Fig. 1) was used to measure drawing speeds up to 30 ft per min and on-off microswitches were used for higher speeds.

Drawing was conducted so that each rod represented one reduction (10, 20, 30, or 40 per cent), one drawing speed (10, 20, 30, or 40 ft per min), and one or more restraining forces (up to 16,650 lb).

The tension specimens are shown in Fig. 2. One smooth and one notched specimen were tested in the drawn condition while the remaining smooth and notched specimens were aged at 400 F for 1 hr prior to finish machining and testing. Both true stress - true strain and engineering tensile data were obtained for the smooth specimens. The notched specimens were tested in a special alignment fixture to insure axial loading.

TABLE 2.—ENGINEERING MECHANICAL PROPERTIES OF SAE 4340 RODS PRETEMPERED AT 1000 F.

Identification[a]	Reduction, per cent	Drawing Speed, ft per min	Back-Pull, lb	Yield Stress (0.2 per cent), psi	Tensile Stress, psi	Reduction of Area, per cent	Elongation, per cent	Notch Tensile Strength, psi
4	...	...	...	176 400	187 900	42.8	13.0	...
4-1A	10	40	16 650	195 400	199 400	42.8	10.0	284 000
4-1C	10	40	16 650	207 400	212 900	39.8	8.0	280 000
4-2A	10	40	9 700	197 400	199 400	44.6	10.0	280 000
4-2C	10	40	9 700	203 400	211 400	39.2	8.0	279 000
4-3A	10	60	0	186 400	196 400	44.0	9.0	274 000
4-3C	10	60	0	186 400	197 900	38.6	9.0	274 000
5	...	...	...	178 900	184 400	45.2	14.0	...
5-1A	20	10	8 100	190 400	198 400	42.8	10.0	278 000
5-1C	20	10	8 100	211 400	217 900	38.6	8.0	282 000
5-2A	20	10	15 800	199 400	205 400	39.8	10.0	284 000
5-2C	20	10	15 800	207 400	214 400	32.8	8.0	280 000
5-3A	20	10	0	188 400	200 400	45.2	11.0	274 000
5-3C	20	10	0	192 400	203 900	41.0	10.0	274 000
9	...	...	...	178 900	188 900	43.4	13.0	...
9-1A	30	4	13 000	207 400	209 400	39.8	8.0	281 000
9-1C	30	4	13 000	221 900	223 900	32.2	7.0	252 000
9-2A	30	10	9 150	210 400	214 400	41.0	9.0	282 000
9-2C	30	10	9 150	215 400	218 900	35.4	8.0	270 000
9-3A	30	10	0	202 400	206 400	38.6	9.0	280 000
9-3C	30	10	0	211 400	215 900	37.8	9.0	284 400
14	...	...	...	173 300	186 400	20.5	8.0	...
14-1A	40	10	0	213 400	221 400	28.1	6.0	272 000
14-1C	40	10	0	227 500	228 500	19.8	5.0	263 500
14-2A	40	10	950	220 400	222 400	25.4	8.0	272 000
14-2C	40	10	950	230 000	231 000	26.0	6.0	...
14-3A	40	10	3 100	218 400	220 400	26.8	6.0	268 000
14-3C	40	10	3 100	226 500	228 500	22.6	6.0	266 000
14-4A	40	43	0	214 400	221 400	28.1	8.0	276 500

[a] A denotes as-drawn; C denotes aged at 400 F for 1 hr after drawing.

One microspecimen was used to examine the rod cross-section and one to examine the longitudinal center line section. Vickers hardness surveys were conducted on the latter.

Test Results

Typical mechanical property data for rods drawn at one speed or more per reduction are presented in Tables 1 to 3.

Effect of Back-Pull:

In Tables 1 and 2, some effect on the ultimate tensile and yield strength is apparent due to the application of back-pull, but a trend is not clearly defined by the data.

The effect of back-pull on the tensile strength of rods pretempered at 800 and 500 F (Table 3) is indefinite; however, the yield strength of the former was increased with back-pull while for the latter there was a definite drop. The latter observation may be associated with residual stresses, as a 400 F aging treatment reversed the trend.

Aging of the as-drawn rods at 400 F

resulted in a definite increase in ultimate tensile and yield properties for all rods independent of the back-pull. An exception to this was noted in Table 2 for a 10 per cent reduction, with no back-pull, of the 1000 F pretempered material. Little or no change due to aging is apparent.

TABLE 3.—ENGINEERING MECHANICAL PROPERTIES OF SAE 4340 RODS PRETEMPERED AT 800 F, 500 F, AND ROOM TEMPERATURE.

Identification[a]	Reduction, per cent	Drawing Speed, ft per min	Back-Pull, lb	Yield Stress (0.2 per cent), psi	Tensile Stress, psi	Reduction of Area, per cent	Elongation, per cent	Notch Tensile Strength, psi
				Pretempered at 800 F				
20	0	0	...	195 400	206 400	41.6	11.0	...
20-1A	10	10	8 150	204 400	217 400	39.8	9.0	267 900
20-1C	10	10	8 150	222 400	230 500	36.6	7.0	210 300
20-2A	10	10	14 800	212 400	219 400	39.2	8.0	259 600
20-2C	10	10	14 800	227 500	234 500	35.4	7.0	227 100
20-3A	10	10	0	198 400	218 400	41.0	10.0	249 500
20-3C	10	10	0	217 400	228 500	37.2	8.0	
				Pretempered at 500 F				
38	0	0	...	226 500	264 500	15.6	6.0	...
38-1A	10	10	8 300	279 600	294 600	32.8	6.0	281 100
38-1C	10	10	8 300	294 600	298 600	21.8	6.0	227 400
38-2A	10	10	12 500	272 500	291 600	26.0	6.0	205 200
38-2C	10	10	12 500	300 600	302 600	23.4	6.0	222 000
38-3A	10	10	0	283 600	290 600	30.2	7.0	215 300
38-3C	10	10	0	291 600	295 600	24.7	6.0	
				Pretempered at Room Temperature				
40-A	0	0	...	222 400	315 600	2.6	3.0	cracked while grinding
40-C	0	0	...	234 100	289 800	21.6	7.1	
40-1A	10	10	8 300	broke in thread				
40-1C	10	10	8 300	broke in thread			7.1	
40-2A	10	10	14 000	broke in thread			7.1	
40-2C	10	10	14 000	369 400	371 000	28.6	6.0	
40-3A	10	10	0	broke in thread			6.0	
40-3C	10	10	0	364 600	366 200	12.4	3.5	

[a] A denotes as-drawn; C denotes aged at 400 F for 1 hr after drawing.

The data on reduction of area and elongation generally indicate a small decrease in magnitude, consistent with the change in strength level for the material pretempered at 1150, 1000, or 800 F. The reverse is true for that pretempered at 500 F or at room temperature and drawn with back-pull where an improved reduction in area is noted as a result of processing.

Effect of Reduction:

Figures 3(*a*) and (*b*) show the average change in conventional tensile properties for the material pretempered at 1000 F as a function of reduction. The improvement in ultimate tensile and yield strength is similar to that pretempered at 1150 F, the primary difference being a slightly greater increase for the latter group.

The greatest increase in tensile properties occurred between 0 and 10 per cent reduction. An additional increase in the tensile and yield strength resulted from the 400 F aging treatment, averaging 5000 to 10,000 psi at each reduction (except 0).

specimens the reduction of area is higher for rod reduced 40 per cent than for the heat-treated rod. The decrease in ductility for the aged material is slightly greater, as would be expected from the higher strength levels.

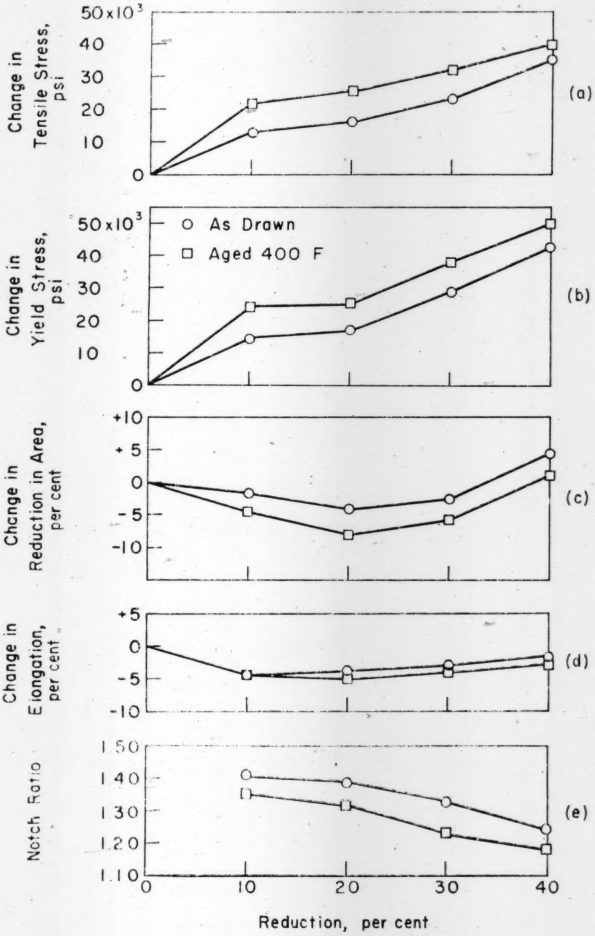


FIG. 3.—Changes in Average Engineering Tensile Properties of SAE 4340 Pretempered at 1000 F.

The decrease in ductility (Fig. 3(*c*)) with increasing reduction up to 30 per cent is consistent with the increase in strength level. However, at 40 per cent reduction the decrease is less than at 30 per cent and in the 1000 F pretempered

Effect of Drawing Speed:

For the range of drawing speeds available, no consistent effect on tensile properties is apparent.

Notched Tensile Strength:

The notched specimen proportions and testing procedure correspond to those recommended for notch toughness screening tests by a special ASTM Committee (10). The notch radius was held to 0.001 in. or less. The notch strength of some specimens exceeds the yield strength by more than the recommended 10 per cent maximum; however, larger specimens could not be prepared because of the constant final diameter of all rods. The notch tensile data were averaged for each reduction and are presented in Fig. 3 for rods pretempered at 1000 F. Other notch data are presented in Tables 1 to 3.

TABLE 4.—AS-DRAWN HARDNESS OF SAE 4340 ROD.
Measurements taken every 0.020 in. starting at the center.

Identification	Pretemper, deg F	Reduction, per cent	Back-Pull, lb	Vickers Hardness										
				Center	0.020 in.	0.040 in.	0.060 in.	0.080 in.	0.100 in.	0.120 in.	0.140 in.	0.160 in.	0.180 in.	Surface
40-2......	room temperature	10	14 000	707	707	707	720	726	733	713	695	672	618	...
40-3......	room temperature	10	0	707	713	733	726	726	726	701	689	677	598	...
38-2......	500	10	12 500	553	557	557	557	557	553	553	553	540	524	...
38-3......	500	10	0	562	562	562	571	566	571	571	557	544	524	...
20-2......	800	10	14 800	435	432	432	432	438	432	435	415	415	404	...
20-3......	800	10	0	423	420	426	432	435	435	429	420	409	399	...
4-1......	1000	10	16 650	390	390	394	394	394	390	387	387	387	390	...
4-3......	1000	10	0	370	366	366	370	370	373	370	363	357	357	...
14-3......	1000	40	3 100	405	409	409	409	409	409	405	405	405	409	...
14-1......	1000	40	0	405	417	409	417	413	413	413	413	417	413	...
24-1......	1150	10	14 850	336	336	339	339	339	336	333	330	336	330	...
24-3......	1150	10	0	330	330	333	333	336	336	333	333	325	312	...
36-2......	1150	40	4 100	360	357	363	360	363	363	363	360	360	357	...
33-1......	1150	40	0	360	360	357	357	360	360	360	357	363	360	...

A decrease in the notch tensile ratio with reduction is apparent for the material pretempered at 1000 F (Fig. 3(*c*)). This decrease is approximately double that for the material pretempered at 1150 F.

Notch tensile ratio curves for the aged condition are lower, but parallel to those for the as-drawn condition. This decrease due to aging is approximately twice as great for the 1000 F pretempered material as for the 1150 F pretempered material, and may be attributed to the higher strength level.

In general, the notch tensile strength reaches a maximum of about 285,000 psi at approximately 210,000 psi tensile strength and 205,000 psi yield strength.

Hardness:

In his review of wire drawing with back-pull, Wistreich (8) noted that there is an increase in hardness toward the center of the wire when drawn without back-pull and that application of back-pull tended to produce a more uniform hardness. Majors (6) observed a uniform hardness in 1-in. diameter 1020 steel rods drawn through a die in a tension testing machine.

The results of this investigation (Table 4) indicate that the hardness tends to become higher and more uniform across the section with (1) increasing reduction and (2) addition of a back-pull. It is not

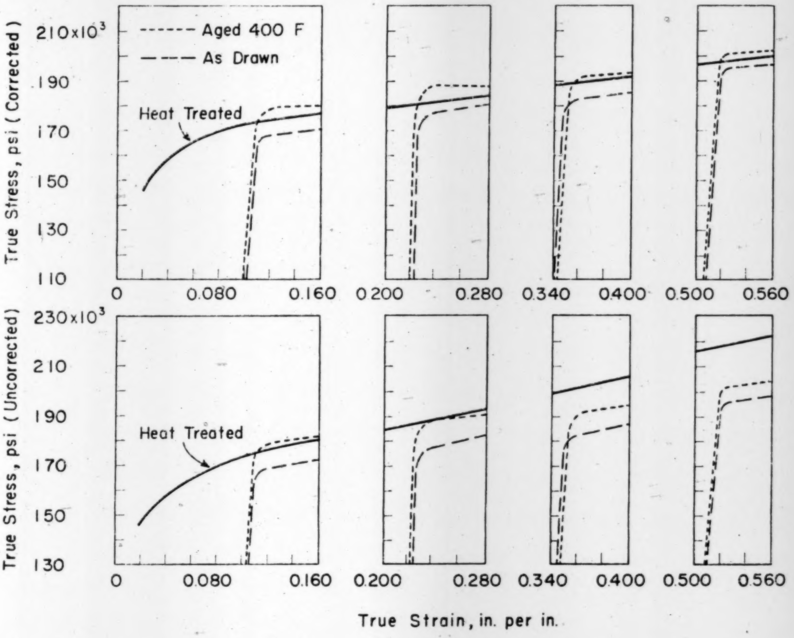


FIG. 4.—True Stress - True Strain Properties of SAE 4340 Rod Pretempered at 1150 F and Drawn 10, 20, 30, or 40 Per Cent Without Back-Pull.

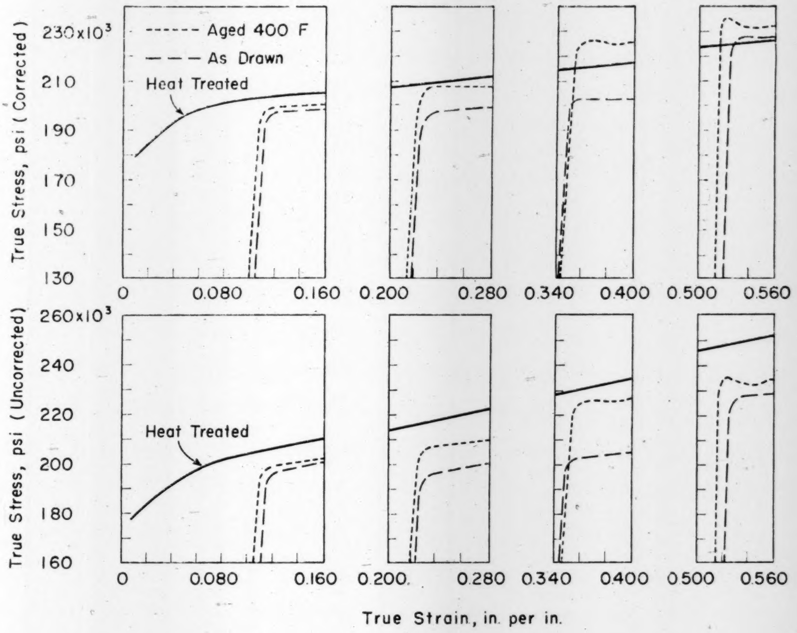


FIG. 5.—True Stress - True Strain Properties of SAE 4340 Rod Pretempered at 1000 F and Drawn 10, 20, 30, or 40 Per Cent Without Back-Pull.

known whether or not this is associated with the stress distribution due to drawing.

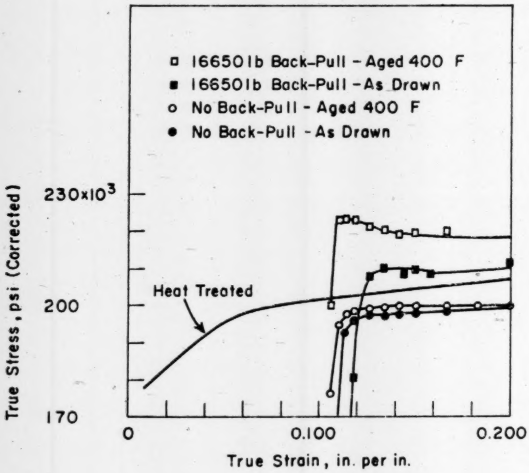


Fig. 6.—Influence of 10 Per Cent Reduction, Back-Pull, and Aging on the Yield of SAE 4340 Pretempered at 1000 F.

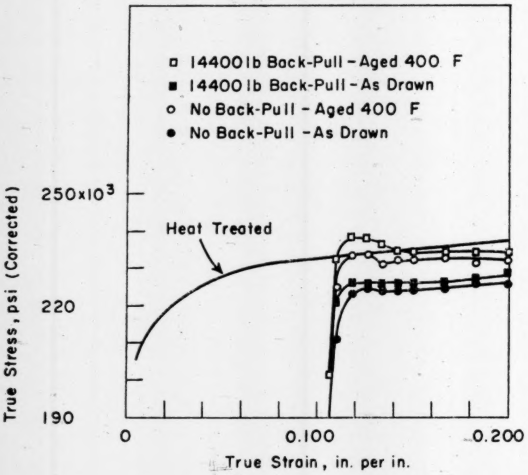


Fig. 7.—Influence of 10 Per Cent Reduction, Back-Pull, and Aging on the Yield of SAE 4340 Pretempered at 800 F.

The hardness is essentially uniform at the center of the rods from which the 0.252-in. diameter tension test specimens were machined. Exceptions to this were the rods pretempered at room temperature which display a hardness maximum at approximately midradius. A surface softening is apparent in several rods pretempered at the lower temperature and may be due to annealing of the surface during the processing.

True Stress - True Strain Properties:

Engineering tensile data are widely used for numerous reasons, among which are the ease with which they may be obtained and adequacy where the elastic region is of primary interest, as in design. However, true stress - true strain tensile data, based on instantaneous measurements of stress and strain, are required as a means of observing the plastic behavior of a metal. In processing, the yield behavior and strain hardening are clearly defined by the true stress - true strain curves and may be related to processing variables.

The true stress - true strain curve has been found to be in error in the region of high plastic strains due to triaxiality in the region of the neck. A method for determining the Bridgman correction for this stress concentration based on the amount of necking strain is described in the Appendix[3] and has been employed in Figs. 4 to 8.

The effect of the correction is readily apparent in Figs. 4 and 5 where corrected and uncorrected true stress - true strain curves are compared for the unstrained and strained rods pretempered at 1150 and 1000 F, respectively. The error due to necking is seen to increase with increasing strain or reduction.

The corrected curves for the as-drawn condition should coincide with that for the heat-treated material at each strain level; however, these are lower in each case. An exception to this is found in Fig. 8 where the flow curve for the unstrained material is lower than that for the strained material. The curve for a duplicate specimen of unstrained material was of the same shape and magnitude as that shown. This may be associated with room-temperature aging.

Regardless of the pretempering temperature, aging at 400 F raised the flow curves to or above that for the heat-treated material. This is true for all of the flow curves with the exception of those for the 10 and 20 per cent reduction in Fig. 5 for rods pretempered at 1000 F. Also apparent in Fig. 5 is the negligible increase in strength level due to aging of the material reduced 10 per cent as compared to the large increase in that reduced 30 per cent. It should be noted that the former was drawn at approximately 60 ft per min and the latter at

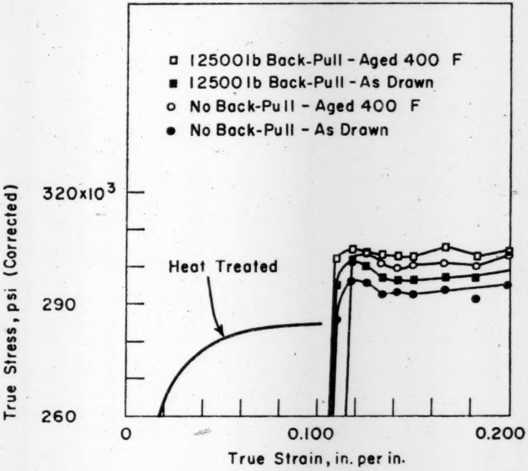


FIG. 8.—Influence of 10 Per Cent Reduction, Back-Pull, and Aging on the Yield of SAE 4340 Pretempered at 500 F.

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[3] See p. 532.

10 ft per min. Another rod, also reduced 30 per cent but at 40 ft per min, nearly coincided with the curve for the heat-treated material; however, the spread between the as-drawn and the aged curves for this rod was negligible. Further study is required to verify these observations.

Aging has little effect on the yield point for reduction up to 30 per cent; however, for a 40 per cent reduction of the rods pretempered at 1150 F (Fig. 4) a sharp yield point has been introduced, and in Fig. 5, for rods pretempered at 1000 F, this is accompanied by an actual drop in the yield.

In Fig. 6 is illustrated another condition under which a significantly higher yield point and a drop in the yield are introduced, that is, the introduction of a back-pull. This was sufficient to raise both the as-drawn and the aged curves for a 10 per cent reduction above the basic heat-treated curve. Aging at 400 F raised the entire curve and emphasized the drop in the yield. This was also noted for the material pretempered at 1150 F and processed similarly, although the effect was not as great.

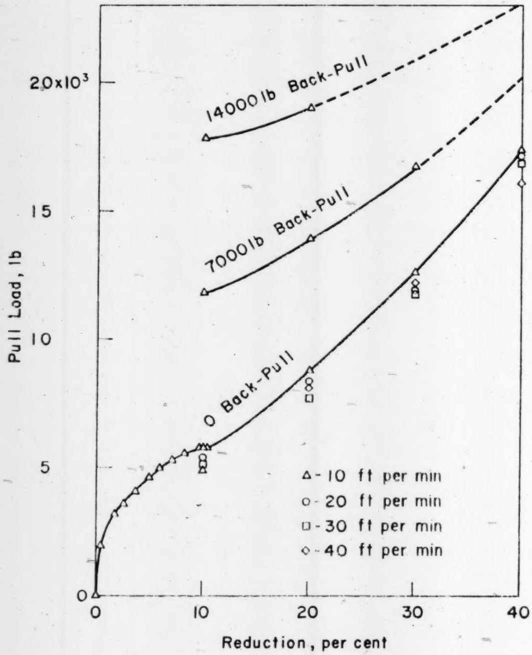

Fig. 9.—Pull Load Versus Reduction for SAE 4340 Rod Pretempered at 1000 F.

In Figs. 7 and 8, for a 10 per cent reduction, an increase in the yield stress and a yield point drop can be seen to be a function of pretempering, reduction, and aging.

Pull Loads:

The effect of back-pull is to increase the pull load by an amount equal to the back-pull as shown in Fig. 9. The total pull load is the sum of the load due to die pressure and that due to back-pull; thus by increasing the tensile force on the rod by means of the back-pull the die pressure required to reduce the cross-section is reduced. The chief advantages of back-pull are decreased die pressures, friction, and wear while the disadvantages include the higher drawing forces and the need for equipment to provide a back-pull.

The effect of the pretempering temperature on the pulling load is shown in Fig. 10 for a 10 per cent reduction. A maximum is apparent for the 500 F pretempered material rather than for the untempered material, as might be expected. This has also been noted in rolling untempered steel strip and is due to the low yield stress of the untempered material.

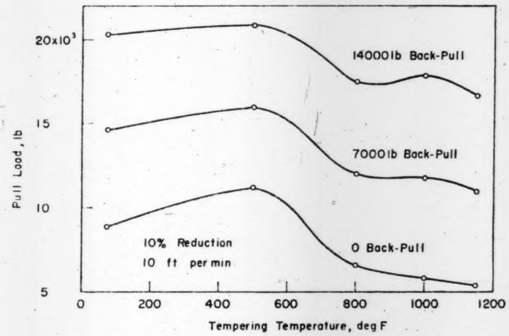

FIG. 10.—Influence of Pretempering Temperature and Back-Pull on Pull Load.

DISCUSSION

Reduction of rods by drawing normally results in an increase in the tensile strength, yield strength, and hardness, and a decrease in ductility as compared to the unstrained material. These effects were apparent in the current work and are principally the result of strain hardening along the standard tensile stress-strain curve. However, it was noted that the strain hardening was not equal in magnitude to that obtained by an equivalent amount of tensile strain. This is shown by the corrected true stress - true strain curves and may be associated with one or more factors.

The difference may be due to an inaccuracy inherent in the method used to determine the Bridgman correction, as the accuracy of the method has not been proved, particularly for high strains.

Another factor may be the difference between the stress systems involved in drawing and in tension testing. The former consists of both a tensile and a compressive stress while the latter consists of tensile stresses only, until necking, after which a triaxial stress system is present. On this basis, a difference in the magnitude of the strain-hardening component may be expected. This is further supported by the results of specimens drawn with back-pull which showed higher strength than without back-pull. The stress system here, with a lower transverse compressive stress and a higher tensile stress, is now like tensile straining.

A temperature effect is present. Essentially no heating is involved in room-temperature tension testing; however, appreciable heating would be expected during the drawing operation, probably to a temperature not exceeding 400 F since a 400 F aging treatment after drawing effectively improved the tensile properties. A higher flow stress curve would be expected for the drawn material due to strain aging.

In addition to the temperature effect, a strain rate effect is also present. Strain rate for the tension test is approximately 17 × 10^{-5} sec^{-1} compared to 5 to 20 sec^{-1} for the drawing. Again, an increase in the flow stress curve would be expected for the latter condition due to the higher strain rate. The fact that the expected increase due to strain aging and strain rate was not observed for material pretempered at 800 F and above may be attributed to a lack of time for all of the potential strengthening due to strain aging to take place during the drawing. This effect is illustrated by Lubahn and Felgar (11) with data obtained by MacGregor and Fisher (12) for SAE 1020 steel tested at 212 F at strain rates of 5 × 10^{-5} sec^{-1} to 500 × 10^{-5} sec^{-1}. An appreciable drop in the flow stress curve is noted with increasing strain rate.

Material pretempered at 500 F may kinetically be more prone to strain aging than the more stable structures formed at higher pretempering temperatures, although the magnitude of the effect may not be too different. For this material the increase in magnitude of the flow stress curve above that expected from the tension test of the unstrained material can be attributed to strain aging during drawing as well as room-temperature aging of the material after drawing.

The pretempering temperature is of particular importance in the strengthening of martensite, as shown in Fig. 11. The increase in strength level for the as-drawn material consists principally of strain hardening. The magnitude of the strain hardening is low for material pretempered at temperatures of 800 F and higher; however, this increases rapidly with decreasing pretempering temperatures below 800 F. A corresponding improvement in ductility is also noted for the material pretempered at the lower temperatures and strained.

A further strengthening increment resulted from the 400 F aging. This appears to be essentially constant and independent of the pretempering temperature. It should be noted that the optimum aging temperature was not determined for each condition and that the optimum aging temperature could increase the strengthening due to aging.

Flow curves for the unstrained mate-

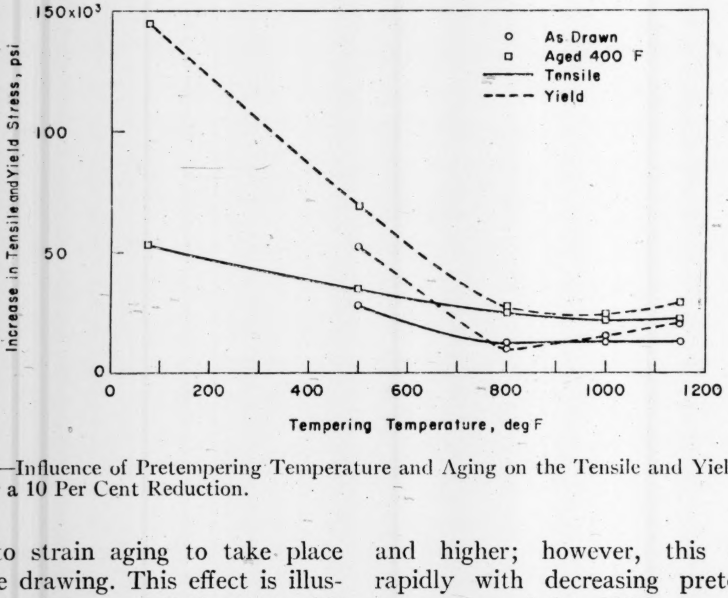

Fig. 11.—Influence of Pretempering Temperature and Aging on the Tensile and Yield Strength Increase for a 10 Per Cent Reduction.

rial do not exhibit a yield point regardless of the pretempering temperature. However, the influence of the pretempering temperature in causing a yield point and a yield point drop in the strained material is appreciable. For material pretempered at 1000 and 1150 F and drawn without back tension, neither a sharply defined yield point nor a yield point drop is noted until a reduction of 40 per cent is achieved. Material pretempered at lower temperatures exhibits a sharp yield for reductions of 10 per cent and, further, the yield point drop becomes more pronounced with decreasing pretempering temperature.

The application of back-pull during drawing has a very potent effect in introducing a sharply defined yield and a yield point drop for a reduction of 10 per cent at all pretempering temperatures including 1150 F.

Regardless of the method by which the yield point phenomena are caused, the effect is greatly enhanced by aging at 400 F, although this is not necessarily the optimum aging temperature.

The notch tensile strength is dependent upon strength level. In this work, a maximum notch tensile strength of 285,000 psi is noted at approximately 210,000 psi tensile and 205,000 psi yield strength. Indications are that other variables investigated, such as pretempering temperature and reduction, may influence the notch tensile strength. For example, for a tensile strength of approximately 220,000 psi attained by a 1000 F pretemper and a 40 per cent reduction, the notch tensile strength is 276,500 psi, but for the same approximate tensile strength attained by pretempering at 800 F and a 10 per cent reduction, the notch tensile strength was only 249,500 psi. Further study of the effects of the variables on the notch tensile strength is required.

CONCLUSIONS

1. Increases in the tensile and yield strength may be obtained by drawing pretempered SAE 4340 rod in the martensitic condition. The increase is due primarily to strain hardening and is dependent upon the pretempering temperature. A further increase, essentially independent of pretempering temperature, results from a 400 F aging treatment.

2. The notch tensile strength is primarily a function of the tensile properties and reaches a maximum at 210,000 psi ultimate tensile and 205,000 psi yield strength. Aging decreases the notch tensile strength.

3. The hardness across the rod cross-section becomes greater and more uniform with (1) increasing reduction and (2) the application of a back-pull in drawing.

4. The magnitude of expected strain hardening based on the tension test stress-strain curve was not achieved in the rods drawn without back-pull. This may be due to (1) an inherent error in the Bridgman correction methods; (2) differences in the stress systems in drawing and in tension testing; or (3) a strain rate effect.

5. A yield point phenomenon evidenced by the appearance of a sharply defined yield point or a yield point drop, or both, was found to be a function of (1) the pretempering temperature; (2) reduction; or (3) back-pull. The effect was increased by aging at 400 F.

6. The pull load required to draw the rods was a function of the rod yield strength (pretempering temperature) and of back-pull.

Acknowledgment:

The author wishes to express his gratitude to E. B. Kula for his many helpful discussions and suggestions, and to J. D. Colgate for his assistance in designing and installing instrumentation on the rod and wire drawing machine and for efficiently conducting the drawing operation.

APPENDIX

METHOD FOR DETERMINING THE BRIDGMAN CORRECTION

The Bridgman correction is expressed mathematically as

$$\sigma_t = \frac{\sigma}{\left(1 + \frac{2R}{a}\right) \ln \left(1 + \frac{a}{2R}\right)}$$

where:

σ_t = corrected flow stress,
σ = measured flow stress,
a = minimum radius, and
R = radius of curvature of the necked region.

Continuous radius measurements are required during the test in order to calculate the correction factors.

A linear relationship between the necking strain, ϵ_n (total strain minus strain at maximum load), and the a/R term in the Bridgman correction was observed by Kula and Larson (**13**) in data obtained by Marshall and Shaw (**14**). This is

$$\sigma_t = \frac{\sigma}{\left(1 + \frac{2}{\epsilon_n}\right) \ln \left(1 + \frac{\epsilon_n}{2}\right)}$$

where $\epsilon_n = a/Rk$ and k was observed to be approximately 1. Nunes (**15**) also investigated the relationship in a recent paper.

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(**10**) Fourth Report of a Special ASTM Committee, "Screening Tests for High-Strength Alloys Using Sharply Notched Cylindrical Specimens," *Materials Research & Standards*, Vol. 2, No. 3, March, 1962, p. 196.

(**11**) J. D. Lubahn and R. P. Felgar, *Plasticity and Creep of Metals*, John Wiley and Sons, Inc., New York, N. Y., 1961.

(**12**) C. W. MacGregor and J. C. Fisher, "Tension Tests at Constant True Strain Rates," *Journal of Applied Mechanics*, Vol. 13, 1946, p. 162.

(**13**) E. B. Kula and F. R. Larson, "Ductility Relationships in Tensile Testing," *Watertown Arsenal Laboratories Report No. 111/24*, Oct., 1957.

(**14**) E. R. Marshall and M. C. Shaw, "The Determination of Flow Stress from a Tensile Specimen," *Transactions*, Am. Soc. Metals, Vol. 44, 1952, p. 705.

(**15**) J. Nunes, "Flow Stress-Strain Relationships in Tension Tests of Steel," *Watertown Arsenal Laboratories Report No. 834.2/10*, March, 1963.

DISCUSSION

Dr. N. N. Breyer[1] (*presented in written form*).—Mr. DiCesare has extended our knowledge of the cold working of tempered and untempered martensitic SAE 4340 steel. It is well understood that the author, in his extensive investigation, could not examine all parameters involved in the thermal-mechanical treatment of martensitic steels. The selection of the reduction in the dies, however, leaves an unanswered question. In the particular case of the material that was either left untempered or tempered at low temperatures, only a 10 per cent draft was used. Was the limitation to this one draft deliberate or were there breaking conditions encountered when higher reductions were attempted?

In the particular case of the untempered martensite, the fact that the author has been able to achieve 10 per cent draft is quite similar to that previously reported in reference (3) of the author's bibliography. However, in this previous work it was found that giving a 10 per cent draft to a 4340 material either resulted in die breakage or in a severely embrittled steel due to the excessive strain hardening. This limitation of per cent deformation was recognized and in the previous work interesting properties were produced with lesser deformations. These lesser deformations permitted high strengths approaching the 400,000 psi tensile strength. It had been found that very little was to be gained by working beyond 5 per cent. The mechanical property improvement for heavier deformations was practically zero and the difficulty of the process was increased. It would be interesting to see if lesser drafts would not have eliminated the erratic results obtained for both the smooth and notched tension tests.

The application of back tension and the resulting more uniform hardness across the section can be explained as proposed by the author by the more uniform tensile mode of deformation. A change in the deformation process might also be reflected in changes of the residual stress patterns in the steel bars. A simple procedure for reflecting the stress pattern in bars has been reported in the literature.[2] The author, by slitting the bar longitudinally through the center, should be able to determine gross differences in the macro residual stress patterns which may be a result of changes in the deformation mode.

Mr. E. DiCesare (*author's reply by letter*).—The author wishes to thank Dr. Breyer for his interesting and helpful discussion. The principal emphasis of this investigation was placed on the lower strength level rods which were tempered at the higher temperatures and drawn 10, 20, 30, and 40 per cent. The 10 per cent reduction was selected for the rods tempered at the lower temperatures or left untempered in order to have a common reduction, 10 per cent, for comparison of all the rods. No other reductions were attempted on this material.

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[1] Manager, Technical Projects, LaSalle Steel Co., Hammond, Ind.

[2] "The Approximate Determination and Thermal Relief of Residual Stresses in Cold Drawn AISI C1060 Steel Bars," Case Institute of Technology, Metals Research Laboratory Technical Report No. 15, *WAL 310/90-61*, Jan., 1952.

The relative magnitude of the longitudinal macro residual stresses was determined on a rod tempered at 1075 F and drawn approximately 10 per cent with backpull of 0, 7000, and 14,000 lb, using the method suggested by Dr. Breyer. Tensile stresses were indicated for all degrees of back-pull, the maximum being present in the section of rod drawn with no back-pull. These were reduced approximately 24 per cent by application of 7000 lb back-pull and approximately 56 per cent by application of 14,000 lb back-pull. The role of back-pull in reducing macro longitudinal residual tensile stresses is apparent.

SUPPRESSION OF THE BAUSCHINGER EFFECT AND CHANGES IN FLOW PATTERN OF DUCTILE METALS CAUSED BY CYCLIC TORSIONAL STRAINS*

BY N. H. POLAKOWSKI[1]

SYNOPSIS

The Bauschinger effect, defined as the difference in yield stresses for continued and reversed straining, essentially disappears after a series of stress cycles of decreasing amplitude. This applies to annealed and initially cold-worked metals alike, although in the latter case the change is accompanied by an over-all decrease of the yield stress (fatigue softening).

The attenuation of the Bauschinger effect also leads to a conspicuous change in the deformation mode of cold-worked (drawn, stretched) metals during subsequent continuous torsion. The ordinarily observed propagation of flow by Lüders-type shear bands recedes or, if the initial work-hardening was moderate, it can be completely suppressed and replaced by homogeneous flow. This is indicative of a relation between the degree of work softening and the development and subsequent stabilization of the Bauschinger effect with increasing prestrain.

The present results are related to the author's earlier work on fatigue softening, and a tentative dislocation mechanism is suggested for both shear band formation and their weakening by cyclic stress. The similarity between the observations here reported and the effects of cyclic stress on the discontinuous yield in iron is pointed out and discussed from the standpoint of the Cottrell-Bilby-Conrad yield concepts.

This paper is concerned with certain permanent changes in the mechanical behavior of metals subjected to large strain cycles of constant or decreasing amplitude. The scope of the work was limited to the prefracture stage and its specific purposes were:

1. To clarify certain basic rules governing the intensity of the Bauschinger effect (B.E.) as a function of plastic strain and the number of cycles.
2. To determine whether the B.E can be suppressed in initially virgin (not work-hardened) metals or in ones subjected to considerable unidirectional prestrain.
3. To attempt to throw additional light on the fatigue softening of cold-worked metals by establishing its relation, if any, to the B.E.

It is appropriate to recall Bauschinger's original observation that a small permanent strain reduced the elastic limit for reversed stressing and that a subsequent application of a cyclic stress could develop new, so-called

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* Presented at the Sixty-sixth Annual Meeting of the Society, Atlantic City, N. J., June 23–28, 1963.

[1] Professor of Metallurgical Engineering, Illinois Institute of Technology, Chicago, Ill.

"natural" elastic limits unrelated to the virgin, or "primitive," elastic range (**1**).[2] Bauschinger's archaic terminology and understandable unawareness of imperfect elasticity manifestations did not detract from the basic value of his findings and conclusions. The work reported here indicates that the latter can be used as a starting point to include situations not anticipated by Bauschinger or those who worked later along similar lines, such as Smith and Wedgwood (**2**).

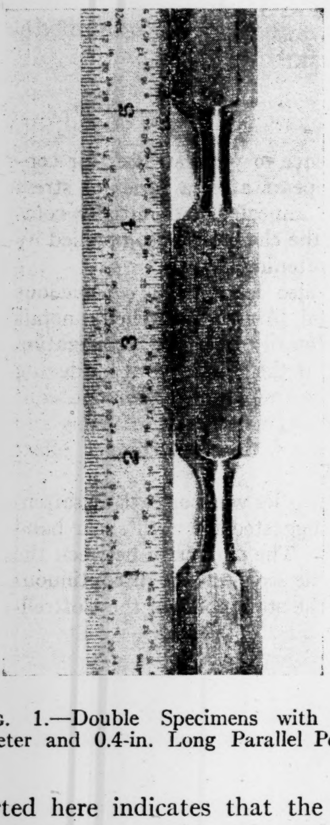

FIG. 1.—Double Specimens with 0.3-in. Diameter and 0.4-in. Long Parallel Portions.

Many have followed up and elaborated on the fascinating phenomenon of fatigue softening of cold-worked metals since it was reported by Palchoudhuri and the author in 1954 (**3**). Several attempts have been made to interpret the gross mechanism or individual facets. However, progress in this respect was not spectacular, because a satisfactory explanation of the Bauschinger effect from the standpoint of the dislocation theory is not yet available (**4,5**). With this in mind, the author proposes to keep his own interpretation of the results reported on a substantially phenomenological level.

EXPERIMENTAL

Materials, Twisting, and Recording:

The experimental materials were selected to represent cases of both a relatively weak B.E. (copper, 95-5 tin bronze) and a strong one (medium-carbon steel) coupled with good ductility (**6**). Specimens were either single or double depending on their purposes. A double specimen is shown in Fig. 1. Slow torsion experiments were performed on annealed and on cold-drawn OFHC copper, 95-5 tin bronze (annealed and cold drawn 30 per cent), 62 per cent drawn steel with 0.016 per cent carbon and 0.49 per cent titanium and on 1045 steel from two different batches. In view of the shortness of the specimens and absence of a torsional strain transducer with a sufficiently short gage length, a time base rather than an *X-Y* recorder had to be used. Except for the unduly reduced slope of the nearly elastic loading portion and the reversed slope of the unloading line, the records were satisfactory. The linearity of the relation between chart lengths and twist angle was very good for a fixed-armature voltage setting of the drive motor. The head speed was about $\frac{1}{2}$ rpm for double and $\frac{1}{4}$ rpm for single specimens.

The diagrams discussed in the following were copied from the charts, omitting the backlash effect during reversal. Also, the unloading lines were drawn vertically in most cases. To avoid

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

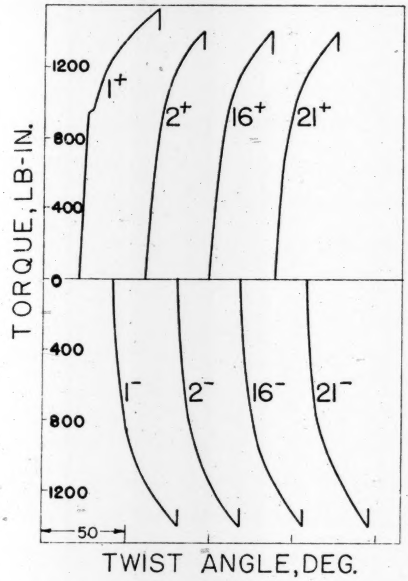

FIG. 2.—Reversed Torsion Records of Annealed 1045 Steel. Cycle number indicated on curves (d = 0.459 in., l = 1 in.).

duplication, only a selection of typical data will be presented.

Cyclic Stressing Experiments:

Figure 2 shows several alternating torsion cycles on the annealed medium-carbon steel. Except for the first half-cycle where a higher torque was applied, all curves are nearly identical. The strain readings indicated, however, a slight but systematic cumulative drift in the direction of the first half-cycle which resulted in a definite residual twist after a number of cycles. The cause for this lies probably in the small but only partially reversible dimensional changes in the specimen which resulted in an effectively higher shear stress in forward twisting compared with reversed half cycles. This drift expectedly decreased with decreasing amplitude, and, in general, it had no effect upon the main argument. In Fig. 3, the suc-

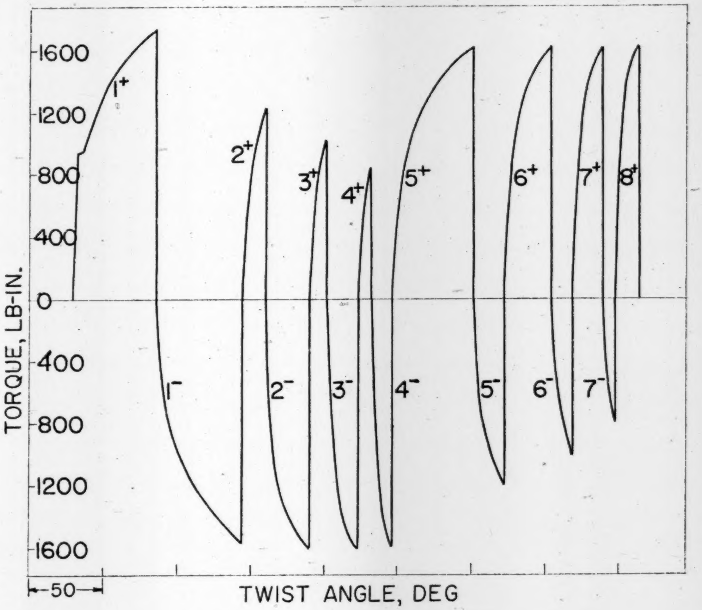

FIG. 3.—Variations of the Bauschinger Effect in Annealed 1045 Steel with Torque Applied Prior to Reversal (d = 0.459 in., l = 1 in.).

cessive torques were gradually reduced in alternate half cycles (2^+, 3^+, 4^+; or 5^-, 6^-, 7^-, 8^-). Whenever the torque was less than its maximum in the preceding half cycle, the B.E. in the following half cycle was weaker. Accordingly, the total subsequent twist angles decreased in the order 2^-, 3^-, 4^-; or 6^+, 7^+, 8^+, respectively, from their maximum values in 1^- and 5^+. If, for instance, curve 7^- had terminated at a torque lower than 800 lb-in., curve 8^+ would have been steeper. If, on the other hand, the ±1600-lb-in. amplitude had been maintained throughout, all the half-cycle curves would have been identical with 5^+.

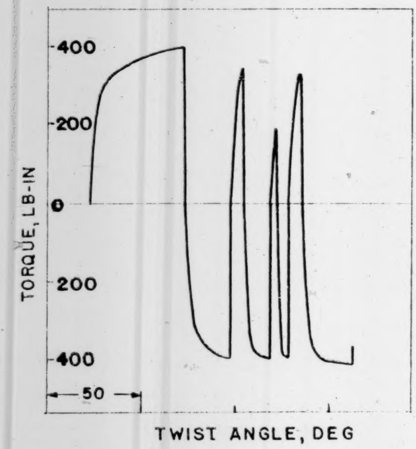

FIG. 4.—Same Effect as in Fig. 3 on Copper (d = 0.415 in., l = 1 in.).

The same effects can be seen on the initially annealed copper specimen in Fig. 4, where the width of the negative half loop decreased with decreasing positive torques. This particular specimen underwent a complex prior strain history which is omitted as inconsequential.

In further pursuing the effects in Figs. 3 and 4, two double specimens (Fig. 1) of 1045 steel were prepared from lightly (18 per cent) cold-drawn hexagon bar and one was annealed at 870 C. Following a large-amplitude first cycle, both specimens received a series of reversed cycles of decreasing amplitude until it appeared that their conditions were approximately elastic. One such cyclic sequence for the as-drawn specimen is reproduced in Fig. 5; for the annealed specimen it was similar but involved 11 full cycles. The two portions of each duplex specimen were then sawn apart

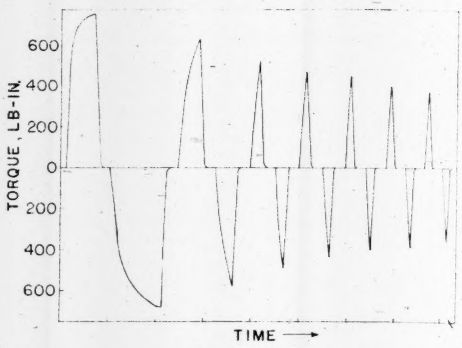

FIG. 5.—Attenuation of the Bauschinger Effect in Cold-Drawn 1045 Steel by Cycling with Decreasing Amplitude (Double Specimen, Fig. 1).

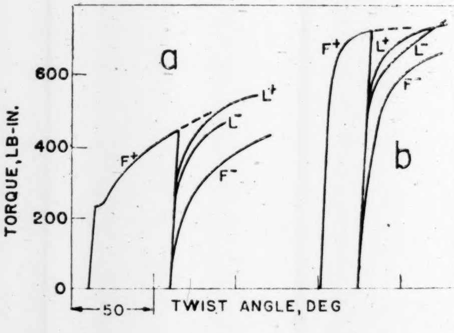

FIG. 6.—Torque-Twist Records in First Cycle (F); After Completed Cycling According to Fig. 5 (L).

L-curves obtained after cutting double specimen in two. Steel 1045 annealed (a) and cold-drawn (b). Symbol (+) is for clockwise twisting, (−) for counterclockwise twisting.

and were continuously twisted in opposite directions, resulting in curves L^+ and L^- (Fig. 6). These final positive and negative curves within each pair were reasonably similar to each other, but neither was a continuation of the respective initial F^+ and F^- curves. Moreover, they fitted between the latter, so that $L^+ < F^+$ and $L^- > F^-$.

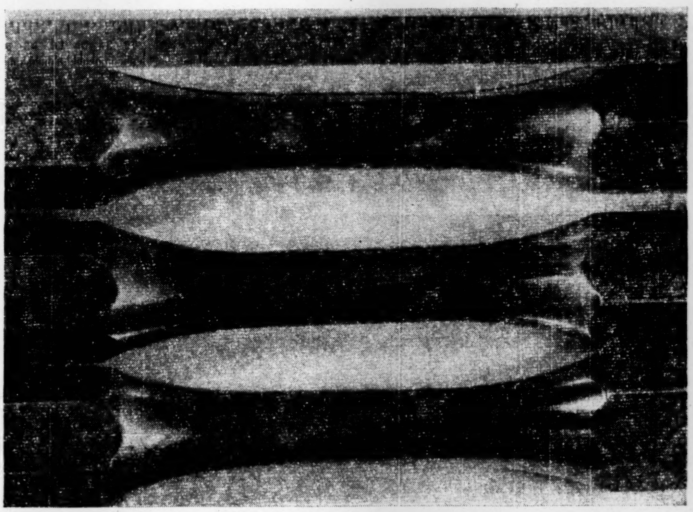

FIG. 7.—Almost Complete Elimination of Discontinuous Flow in Unidirectionally Twisted 30 per cent Cold-Drawn Bronze By Several Cycles of Reversed Torsion. (*Top*) As-drawn; (*middle*) after three cycles; and (*bottom*) after eight cycles. Heavy India ink line along specimen was straight prior to final twisting.

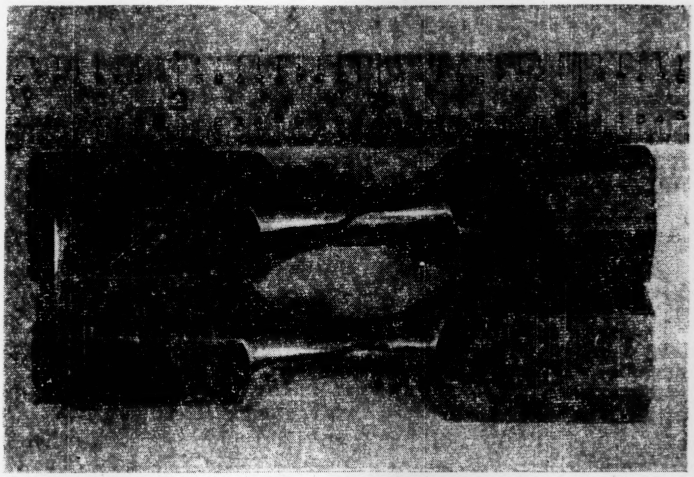

FIG. 8.—Effect of Plastic Torsion Cycles on Twisting of 57 per cent Drawn Copper: (*top*) Before Cycling; (*bottom*) After Cycling.

In plastic cycling experiments on heavily cold-worked bars, formation of discontinuous shear bands (**7**) must be avoided. Once such a band was allowed to develop, subsequent cyclic strains were confined to it while leaving the remainder of the specimen unaffected. In these conditions, the maximum uniform cyclic strain was limited to a small strain, γ_u, which preceded the onset of severe localized shearing. This pre-yield strain was much smaller in the steels than in copper and its alloys.

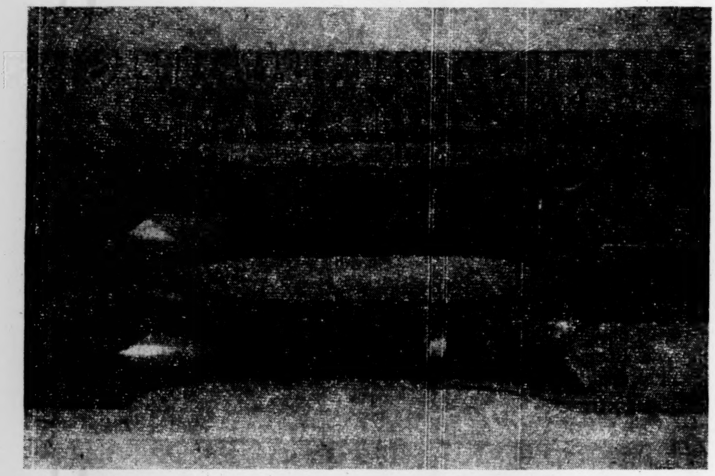

FIG. 9.—Same Effect as in Fig. 8 on 62 per cent Cold-Drawn Titanium Steel: (*bottom*) Before Cycling; (*top*) After Cycling.

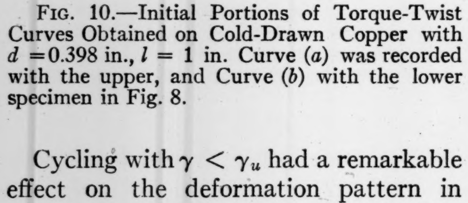

FIG. 10.—Initial Portions of Torque-Twist Curves Obtained on Cold-Drawn Copper with d = 0.398 in., l = 1 in. Curve (*a*) was recorded with the upper, and Curve (*b*) with the lower specimen in Fig. 8.

Cycling with $\gamma < \gamma_u$ had a remarkable effect on the deformation pattern in subsequent unidirectional torsion. The tendency to form localized shear areas gradually receded and the bands became progressively less pronounced as the number of prior cycles increased (Figs. 7 to 9). When the initial work-hardening level was moderate, formation of localized bands could be virtually suppressed (Fig. 7). In heavily drawn bars the bands merely became fainter and diffuse compared with the as-drawn condition, but never disappeared completely (Figs. 8 and 9).

The changes of flow modes in the sequences shown in Figs. 7, 8, or 9 were accompanied by corresponding changes in the stress-strain (torque-twist) relations, which followed the pattern set by curves F^+ and L in Fig. 6(*b*). The yield stress fell while the slope of the post-yield curve increased with progressive cycling, as typically illustrated by the sequence in Fig. 10.

Large cyclic strains developed rapidly an increasing surface roughness in all cases. This was followed by multiple cracking, which started on longitudinal rather than transverse maximum-shear planes, especially in the harder 1045 steel, a fact described in more detail elsewhere (8).

DISCUSSION

The Universal Validity of Bauschinger's Rule:

Bauschinger formulated the rule that a tensile (or compressive) stress beyond the elastic limit reduces considerably the elastic limit in compression (or tension) the higher the applied stress is above the original elastic limit (**1**). This rule is long known to apply to strains from a fraction to several per cent, and the B.E. is very spectacular when the primitive yield strength is relatively high (**9–11**). At still larger prestrains or when the primitive yield stress is inherently low (aluminum, copper), the B.E. is best defined as the difference between the yield stresses for continued and reversed straining because the initial work-hardening raises the yield stress with respect to both directions. This definition was proposed by Sachs and Shoji (**10**) as a result of their experiments with brass single crystals.

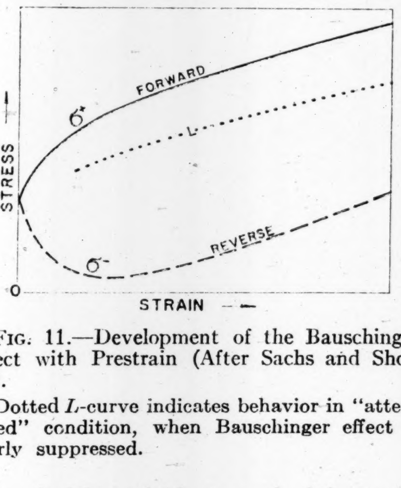

FIG. 11.—Development of the Bauschinger Effect with Prestrain (After Sachs and Shoji (9)).

Dotted *L*-curve indicates behavior in "attenuated" condition, when Bauschinger effect is nearly suppressed.

The B.E. appears with comparable intensity in single and polycrystals (**10,11**). There is no evidence to indicate that it is greatly affected by residual body stresses, and it is equally strong in tension-compression as in alternating torsion of solid specimens with their inherently high residual shear stresses.

Suppression of the B.E. by Attenuated Stress Cycling; Nondirectional Work-Hardened State:

The variation of the B.E. with prestrain is illustrated schematically in Fig. 11 using the Sachs-Shoji plot. The upper curve (σ^+) is the stress-strain relation for unidirectional deformation. The lower, dashed σ^- curve depicts the reversed yield stress at any forward prestrain. The ordinate distance between the two represents the B.E.

Turning to the final torsion curves in the two groups in Fig. 6, it is assumed that L^+ and L^- in each pair are identical except for the sign. They can thus be replaced by a single, representative curve *L*. The actual difference between the two is probably due to secondary factors and is of little significance for the argument that follows. Such an *L*-curve is clearly intermediate between the dashed continuation of the first half-cycle F^+ and the conventional Bauschinger curve F^- obtained in the first strain reversal.

From Figs. 3 through 6 it follows that a tapering-off cyclic-stress range shifted the "forward" curve down and the "reverse" curve up. When they merged into a single *L*-curve as explained above, the metal was obviously work hardened but devoid of directional differences in resistance to deformation in at least two opposite directions. Curve *L* was located in Fig. 11 arbitrarily just to illustrate this point.

Since there is no known way to separate the plastic B.E. and the hysteresis loop component due to anelasticity, one cannot be certain whether the B.E. can ever be totally eliminated (although it certainly can for most practical purposes). This is particularly true of metals that do not have a true fatigue limit. If the latter exists, however, it must lie on a Bauschinger's "natural elastic limit" locus, curve *L*.

Static Deformation of Fatigued Metals:

The initial yield stresses, the maximum alternating stress range, and the resulting *L*-curves in Fig. 6 are apparently unrelated. The same is true of conventional fatigue and the associated small strains, at least in the stress ranges where fatigue failure is certain to occur. Although published data refer only to one-way static tests (tension or compression but not both on the same material (3,12–15)), it is conspicuous that in no case was the static yield stress of a fatigued metal equal to the maximum cycle stress. The elastic-plastic transition was gradual, and no characteristic value could be assigned to it except as an arbitrary offset strain. This applies to annealed and cold-worked metals alike.

Discontinuous Flow (Yielding) and Its Suppression by Alternating Strain; Prior Cold Work Versus Interstitial Locking:

A shear band, progressively spreading along a cold-worked bar subjected to plastic torsion, immediately brings to mind the propagation of a single Lüders band in iron. The similarity is further accentuated by the fact that both spread under a constant stress, Fig. 10(*a*). If some bands stop and new ones start, the stress fluctuates up and down, as shown in Fig. 8 of reference (7), in connection with torsional shear bands but which needs no illustration insofar as discontinuous yielding of polycrystalline iron is concerned.

These phenomenological similarities suggest that the underlying atomic-scale mechanisms could well be analogous or even identical in both cases. It seems that Conrad's modification of the Cottrell-Bilby yield point theory might be considered for this purpose (16).

While retaining the idea that most of the dislocations are immobilized by interstitial atoms, Conrad attributed the abrupt yield to a cataclysmic multiplication by cross-slip of a small number of free dislocations which must be there to account for the pre-yield strain. In the present case, however, a carbon or nitrogen "atmosphere" must be ruled out inasmuch as copper, non-aging titanium steel, and ordinary mild steel (7) behave alike. The obstacles to a free passage of gliding dislocations must apparently be a result of the initial, pre-torsion work-hardening process itself.

Work hardening is currently attributed to an accumulation of lattice defects such as sessile dislocations, dragging jogs, and pile-ups of dislocations against structural barriers. They all form a three-dimensional network stabilized by interaction forces between the stress fields of the individual defects. This defect structure possesses distinct directional properties, and its resistance to slip varies accordingly.

If the system of external forces is radically changed after preliminary cold working, new slip systems are set in motion. However, the newly activated dislocations soon run up against the already existing defect structure. For the new slip to proceed, the operative dislocations must be forced through the "walls" of this network or bypass the existing barriers. This occurrence is preceded by a modest plastic strain, γ, until, at $\gamma = \gamma_u$, sufficient pressure is built up upon the dislocations piled up in front of the obstacle walls to finally effect a breakthrough at some location, probably by means of the ubiquitous Orowan-Koehler double cross-slip.

The responses to alternating strains of steels with a yield discontinuity on the one hand and of a prestrained ductile metal on the other are also similar, at least in their external manifestations, since the yield stress is depressed in either case. The breakup of the original rigid tie-up proceeds probably through creation of microstress gradients.

In the cases displayed in Figs. 7 to 9, the to-and-fro strains must have resulted in a progressive breakup of the ties between the temporarily immobilized dislocations, a dispersion of some of the pile-ups, and, possibly, an annihilation, by recombination, of many of the now unlocked dislocations. The required microstress gradients could arise by a gradual escape of some of the dislocations in pile-ups by cross-slipping, a process held responsible for fatigue softening by Ham and Broom(5) who found that the minimum cyclic stress for fatigue softening coincides with the beginning of slip-band extrusion. Whether cross slip is indeed the principal mechanism for such extrusion is not yet a certainty, although it is quite plausible. Irrespective of these discrete details, however, all the above-named factors should jointly result in a decrease of the mean-lattice strain and the latent strain energy associated with it (fatigue softening (3,5,14,15,17)).

In a typically yielding steel, the shortening of the yield platform and the associated fall of the yield stress can be attributed to a step-by-step pulling away of dislocation segments from locking interstitials. If the matrix is soft (annealed), the microstress gradients will be created by work hardening rather than softening, however.

Whereas cross slip would seem to be an essential element for both cyclic softening and unidirectional flow in torsion after a static prestrain, continuous deformation after cycling can probably proceed by both simple and cross slip with equal ease.

Regeneration of Ductility by Alternating Strain:

The gradual increase of elongation to fracture of cold-worked metals following alternating strains of increasing number or amplitude (18) is a direct consequence of the changes in the stress-strain curve (Fig. 10). Owing to the increase of the strain-hardening exponent from situation (*a*) to (*c*), as demonstrated by the rounding of the yield region and the increasing average slope, the necking point is shifted to the right. The gain of ductility is then due to increasing uniform elongation.

Relation Between the Intensity of Shear Bands After Cycling and the Bauschinger Effect:

It has already been pointed out that localized shear-band formation could be virtually suppressed when the static prestrain was moderate but only weakened after severe prior deformation (Figs. 7 to 9). This suggests a relation to Fig. 11 where the fall of yield stress on account of the B. E. (that is, the difference between the σ^+ and σ^- curves) became nearly constant to the right of the minimum on the lower curve. The above coincides with an earlier observation according to which the fall of indentation hardness after reversed deformation becomes constant in a given material beyond a certain initial prestrain (6). It would appear, therefore, that the fall of yield stress, the fall of indentation hardness, and the weakening of the shear-band intensity in the unsafe fatigue range are all related in some way, perhaps even quantitatively, to the inherent intensity of the B.E. in a particular metal. It is expected that future work based on the findings described here will supply details of this relationship.

CONCLUSIONS

1. Alternating stress cycles in the plastic range produce substantially closed stress-strain loops and a strongly developed Bauschinger effect.

2. A gradual decrease (attenuation) of the cyclic range to a level at which the

plastic strains become negligibly small results in a condition where the unidirectional stress-strain curves in subsequent static tests are nearly identical for both forward and reverse straining.

3. The above treatment thus effectively suppresses the Bauschinger effect and, consequently, leads to a nondirectional work-hardened condition. This is true of a variety of body-centered and face-centered cubic metals and alloys, whether initially workhardened or not.

4. Statically cold-worked (drawn, stretched, rolled) ductile metals deform in torsion nonuniformly, by propagation of shear bands. When an attenuated cyclic treatment is applied to such material, the tendency to deform nonuniformly is greatly reduced or even eliminated, depending on the level of initial work-hardening.

5. This change in the severity of the shear bands is accompanied by pronounced work-softening (fatigue softening).

6. There are a number of striking and probably significant similarities between the aforementioned phenomenon and the gradual annihilation of discontinuous yielding in soft iron and steel by cyclic strain. It is likely, therefore, that the formation mechanisms of shear bands in torsion and of Lüders bands are similar also on an atomic scale.

Acknowledgment:

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FACTORS INFLUENCING TENSILE AND NOTCH-TENSILE PROPERTIES OF HIGH-STRENGTH STEELS

By E. P. Klier,[1] B. B. Muvdi,[2] and George Sachs[3]

Synopsis

The results of tension and notch-tension tests are presented for 4340, 4330 V-modified, 98B40, and four proprietary high-strength steels. The steels were heat treated to characteristic strength levels in the range from about 180,000 to 300,000 psi. Longitudinal and transverse tension and notched tension specimens to 0.9-in. diameter were tested. The different diameter notch specimens were notched to give elastic stress concentration factors of $K = 3, 5,$ or 10.

In general, the tensile strength was found to be independent of the specimen orientation but to decrease gradually with increase in specimen size. The ductility of smooth specimens was observed to depend on both specimen orientation and specimen size.

The notch strength decreased with increase in stress concentration, specimen diameter, and as-processed section size. It also decreased as the specimen orientation was changed from longitudinal to transverse. As the tensile strength was reduced to less than about 200,000 psi these effects became of little significance.

The static tensile data for smooth and notched specimens presented in this report constitute an augmentation and extension of data presented earlier (**1**).[4] The effects of a number of variables on the tensile properties of high-strength steels have been more thoroughly evaluated and this paper includes the results of studies of (1) specimen orientation, (2) as-tested section size, (3) notch sharpness, (4) strength level, and (5) composition. Seven steels were tested, namely, 4340, 4330 V-modified, 98B40, 300 M, Super Hy-Tuf, Hy-Tuf, and Super TM-2.

Experimental Procedure

Materials:

The steels were in the form of hot-rolled sections from commercial electric furnace heats. The chemical analyses and section sizes are presented in Table 1.

Test Specimen:

The tension and notch-tension specimens are presented in Fig. 1. These were rough machined to 0.015 in. oversize on each surface and were finished after heat treatment. Smooth specimens were finish ground, while notches were machined to the dimensions given in Table 2 by means of a properly contoured carbide tool. The specimens were tested in the fixture described by Klier and Weiss (**2**).

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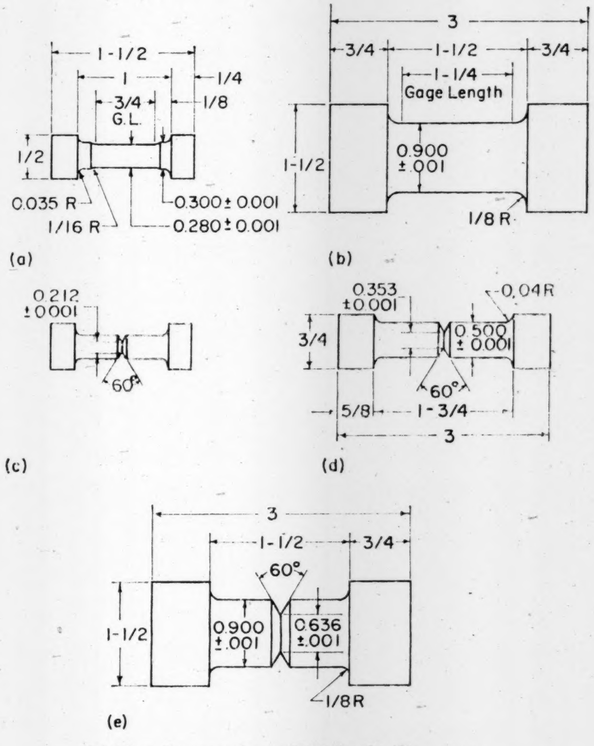
[3] Syracuse University, Syracuse, N. Y.; now deceased.

[4] The boldface numbers in parentheses refer to the list of references appended to this paper.

TABLE 1.—COMPOSITION AND SIZE OF THE VARIOUS STEELS INVESTIGATED

Alloy	Size and Shape	Alloying Elements, per cent									
		Carbon	Manganese	Phosphorus	Sulfur	Silicon	Nickel	Chromium	Molybdenum	Vanadium	Copper
4340	$4\frac{1}{4}$ in. round	0.41	0.79	0.013	0.016	0.31	1.83	0.77	0.23	...	...
4330 V-modified	4 in. square	0.32	0.88	0.012	0.018	0.26	1.79	0.84	0.355	0.07	...
98B40	$4\frac{1}{2}$ in. round	0.46	0.79	0.017	0.017	0.35	0.86	0.81	0.19	...	...
300 M	$4\frac{1}{4}$ in. square	0.39	0.74	0.014	0.014	1.54	1.83	0.83	0.38	0.07	...
Super Hy-Tuf	3 in. round	0.41	1.28	0.014	0.024	1.77	...	1.26	0.33	0.17	...
Hy-Tuf	3 in. round	0.285	1.29	0.019	0.015	1.58	1.87	0.24	0.40	...	...
Super TM-2	3 in. round	0.41	0.72	0.012	0.014	0.61	2.08	1.15	0.44	...	0.14


1-1/2
1
1/4
3/4
1/8
G.L.
1/2
0.035 R
1/16 R
0.300 ± 0.001
0.280 ± 0.001
(a)
3
3/4
1-1/2
3/4
1-1/4
Gage Length
1-1/2
0.900
±.001
1/8 R
(b)
0.212
±0.001
60°
(c)
0.353
±0.001
0.04R
3/4
0.500
± 0.001
60°
5/8
1-3/4
3
(d)
3
1-1/2
3/4
60°
1-1/2
0.900
±.001
0.636
±.001
1/8R
(e)

(*a*) Tension specimen 0.28 in. in diameter.
(*b*) Tension specimen 0.9 in. in diameter.
(*c*) Notch tension specimen 0.3 in. in diameter.
(*d*) Notch tension specimen 0.5 in. in diameter.
(*e*) Notch tension specimen 0.9 in. in diameter.

FIG. 1.—Test Specimens.

TABLE 2.—STRESS CONCENTRATION FACTORS AND CORRESPONDING NOTCH RADII FOR NOTCH-TENSION SPECIMENS (3).

Unnotched Diameter, in.	Notch Diameter, in.	Notch Root Radius, in.	K
0.300......	0.212	0.0110	3
		0.0035	5
		0.0008	10
0.500......	0.353	0.0180	3
		0.0060	5
		0.0013	10
0.900......	0.636	0.0342	3
		0.0100	5
		0.0023	10

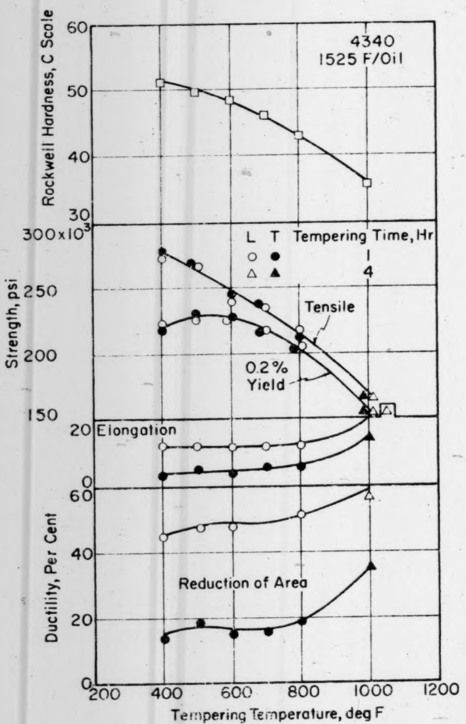


FIG. 2.—Hardness, Strength, and Ductility of 0.28-in. Diameter 4340 Steel Specimens as Functions of Tempering Temperature.

The specimens were heated to the recommended austenitizing temperature for each steel. The 0.3- and 0.5-in. diameter specimens were maintained at this temperature for 1 hr and the 0.9-in. diameter specimens for 2 hr. The specimens were oil quenched and tempered for 4 hr and air cooled.

EXPERIMENTAL RESULTS:

A. PROPERTIES OF 4340 STEEL

4340 steel was investigated more thoroughly than the other steels and the results for this steel are discussed in detail. In general, the conclusions drawn from the tests on 4340 steel apply qualitatively to the other alloy steels investigated.

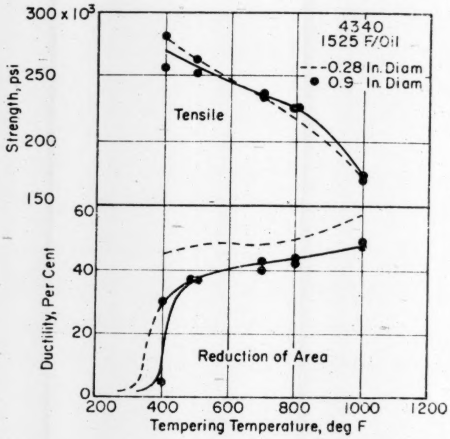


FIG. 3.—Strength and Ductility of 4340 Steel as Functions of Tempering Temperature for 0.9-in. Diameter Specimens.

Effects of Directionality on Tensile Properties:

Figure 2 presents the tensile properties and hardness of 4340 steel for small specimens in both the longitudinal and transverse directions. The yield and tensile strengths in both directions were the same. The reduction of area depends strongly on orientation, and the difference between longitudinal and transverse ductility is large and remains nearly constant at lower tempering temperatures. However, this directionality effect decreases considerably as the tempering temperature increases above about 900 F, corresponding to a tensile strength of about 190,000 psi. In general this conclusion is in agreement with earlier test data (4).

Effect of Section Size on Tensile Characteristics:

The effect of section size on the properties of smooth specimens has not yet been fully evaluated. The tensile data given in Fig. 2 relate to specimens having a diameter of 0.28 in. The results of tests on smooth 0.9 in. diameter 4340 steel test bars are given in Fig. 3, with the trend lines for the 0.28-in. diameter specimens indicated. Only the tensile strength and reduction of area (ductility) are plotted; the yield strength and elongation were also determined but are not reported because of the short gage length of the specimen.

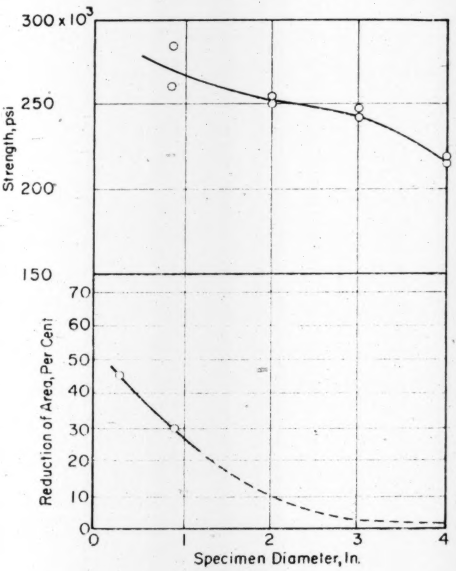


FIG. 4.—Tensile Properties of 4340 Steel Tempered at 400 F as Functions of Specimen Diameter (From Schmidt (6)).

Figure 3 illustrates that the tensile strength of the two section sizes agreed within the limits of accuracy. However, the ductility (reduction of area) is found to decrease, in most instances, by 10 ± 5 per cent as the section diameter increases from 0.28 to 0.9 in. Such an effect is in agreement with previous test data (5).

As the test section size is allowed to increase to large dimensions as compared to the maximum size that could be tested in this investigation, and when the 4340 steel is tempered at 400 to 450 F a profound reduction in both ductility and strength is observed (Fig. 4) (6). In the 4-in. diameter sections the tensile strength is reduced from the expected value of 270,000 to 300,000 psi to about 220,000 psi, a maximum reduction of 20 per cent. At the same time the reduction of area falls to a very low value, and there is an obvious section size effect.

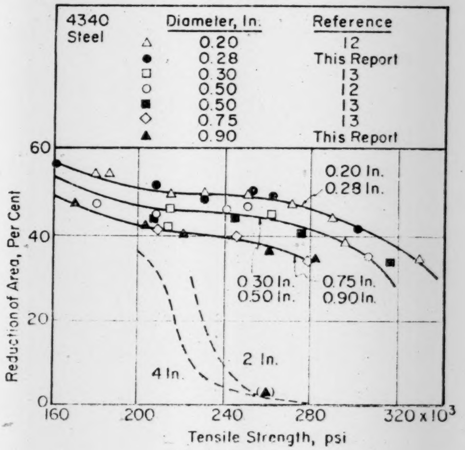


FIG. 5.—Relation Between Tensile Strength and Reduction of Area for Different Test Sections of 4340 Steel.

These data and the embrittling trend associated with increase in section size are further revealed in Fig. 5, where reduction of area is plotted against the tensile strength as established for small specimens. For the larger sections trend lines are projected, but these are hypothetical; while they are qualitatively correct and quantitatively are probable, no direct evidence is available for their construction.

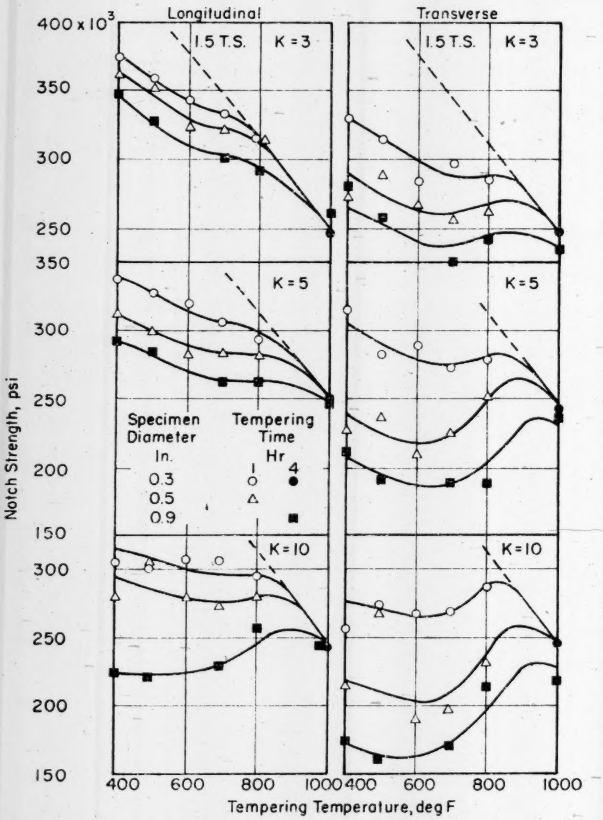


FIG. 6.—Notch Strength of 4340 Steel as a Function of Tempering Temperature with Specimen Diameter as Parameter.

The section size effect established may be due to heat-treating effects of a metallurgical nature, to mechanical factors, or to an interaction of both. A number of factors have been considered as leading to the behavior indicated, including slack quenching (6) and residual stresses (7).

In measurements of stresses developed during still oil quenching, it has been shown (7) that 4340 steel at sizes up to about $1\frac{1}{2}$ to 2-in. diameter forms surface tension stresses. At greater diameters these surface stresses become compressive in sign. In magnitude they are as great as 100,000 psi after quenching, but they are significantly reduced on tempering. After tempering at 450 F in a 4-in. round 4340 steel specimen, tensile stresses ≈40,000 psi have been measured (7) toward the center of the bar. These data, however, do not possess good reliability and require confirmation. With reference to their possible effect on the fracture behavior of the large tension bars, there can be little question of their importance. Since the fracture in the large bars is initiated in the center of the test section, any residual tensile stress will reduce proportionately the load-carrying ability of the section. A 40,000-psi residual tensile stress in the center of the test bar will lower the fracture strength in this section to about 240,000 psi. With the introduction of slack quenching products at the same location, it would seem plausible that the breaking strength be somewhat further lowered.

One final factor is introduced, after which resolution of the question will be left open. The breaking process is not subject to the law of similitude (8). Because of this, for full analysis of the fracture process detailed information would be required for the fracture nucleus and its development for the large and small specimens. That this term is important is readily apparent from consideration of the reduction of area term in Fig. 5.

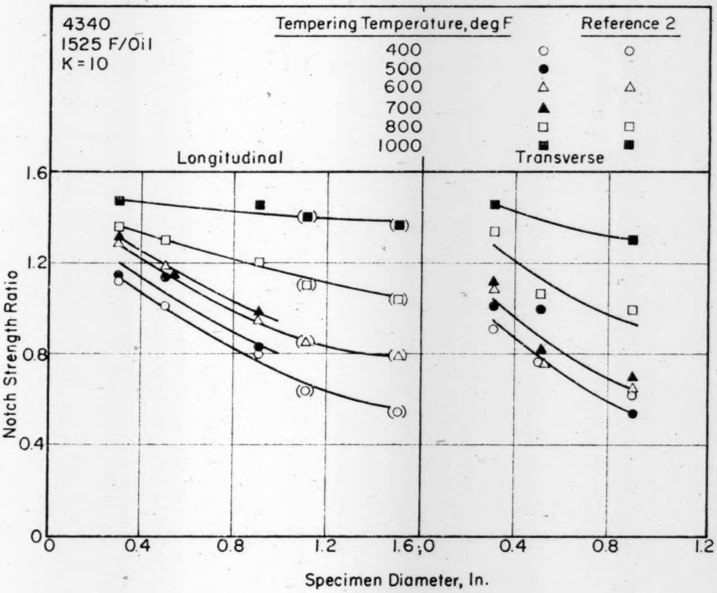


FIG. 7.—Notch Strength Ratio of Sharply Notched ($K \geq 10$) 4340 Steel Specimens as a Function of Specimen Diameter.

Effects of Directionality on Notch Strength:

The notch strength of a heat-treated steel has been observed earlier to depend on the direction of loading relative to the fiber direction (5). The results presented in Fig. 6 for 4340 steel indicate that specimens taken parallel to the direction of rolling (longitudinal) generally possess a notch strength superior to that of specimens taken perpendicular to the direction of rolling (transverse), when heat treated to strength values above 200,000 psi. The magnitude of this effect remains approximately constant for tempering temperatures between 400 and 700 F (see Fig. 6). As the temperature further increases, the transverse notch strength gradually approaches and equals the longitudinal notch strength, after tempering at 1000 F. However, it is evident from

Fig. 6 that the effect of tempering temperature is different for longitudinal and transverse specimens at high strength levels. The transverse specimens are characterized by a pronounced reduction in notch strength.

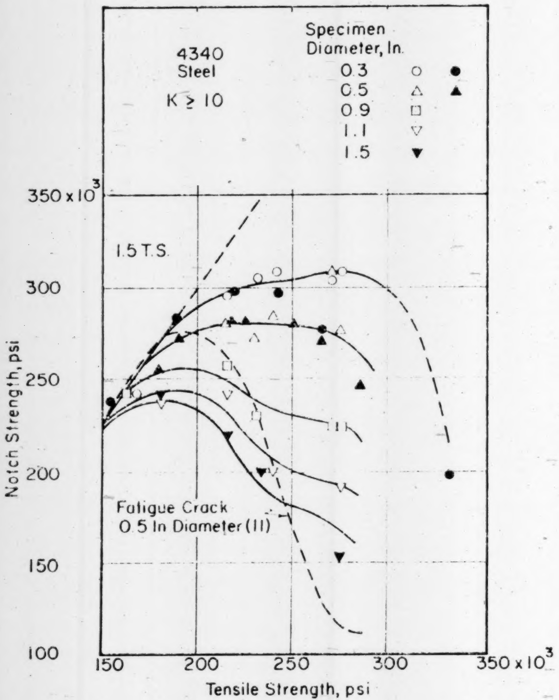


FIG. 8.—Notch Strength of Sharply Notched ($K \geq 10$) 4340 Steel Specimens as a Function of Tensile Strength with Specimen Diameter as Parameter.

Effect of Section Size on Notch Strength:

A change in section size from 0.3 to 0.5 in. leads to reduced notch strength at high strength levels (1,2,5). As the tensile strength of the steel is reduced, this notch strength reduction with size increase is reduced and becomes unimportant at a strength level of about 180,000 psi for 4340 steel (2). The values of notch strength of 4340 steel for the three specimen sizes investigated are presented in Fig. 6 as a function of tempering temperature. This graph clearly reveals the pronounced reduction in the notch strength of the high strength levels with increasing section size. As the tempering temperature increases, the section size effect progressively decreases. On tempering at 800 F, the section size effect is in part eliminated for longitudinal specimens, while it is not present after tempering at 1000 F. For transverse specimens, the section size effect is greatly reduced for the high tempering temperature conditions.

The dependence of notch strength ratio[5] on the specimen diameter is illustrated in Fig. 7 for both longitudinal and transverse specimens with a stress concentration $K = 10$. Values for lower stress concentrations were generally found to respond in a manner similar to those of $K = 10$. The results presented in Fig. 7 clearly reveal that the sensitivity to notching is enhanced with increase in the specimen diameter and decrease in the tempering temperature as discussed previously. Furthermore, this effect is observed to be more pronounced with transverse than with longitudinal specimens. Because of the large scattering of the measured transverse tensile strength, the transverse notch strength ratios were computed using longitudinal tensile strength measurements.

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[5] Notch strength ratio is the ratio of strength of notched specimens to that of smooth specimens.

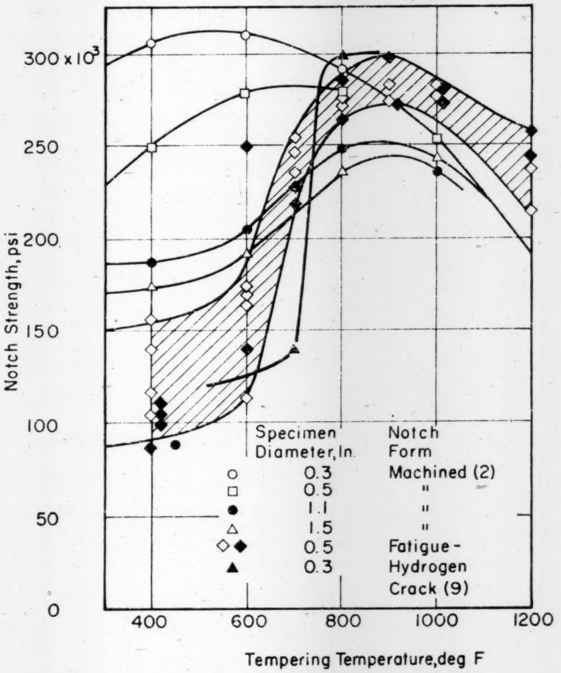


FIG. 9.—Notch Strength of 4340 Steel as a Function of Tempering Temperature for Sharply Notched Specimen, with Specimen Diameter as Parameter, Tested at 75 F.

Figure 8 includes data obtained in previous investigations for large as-tested sections (1,2,5). These illustrate the general agreement between the results of various studies of the notch strength of 4340 steel.

Effect of Stress Concentration:

In the earlier report (1) the notch strength of high-strength steels was determined for calculated elastic stress concentration factors. This requires a progressive enlargement in notch radius as the section diameter is increased. This root radius increase despite the calculated constancy in K could be interpreted to give a less severe notch. Despite this the notch strength in the largest specimen is relatively reduced compared to the smaller specimens for comparable elastic stress concentration factors (Fig. 9). The transverse specimens are again more sensitive to the action of notches, and this sensitivity is only partially lost after tempering at 1000 F. The data in

Fig. 9 emphasize the nature of size effect in notched specimens and indicate that this factor is regularly to be expected to influence the materials properties when the heat-treated structure is notch-sensitive.

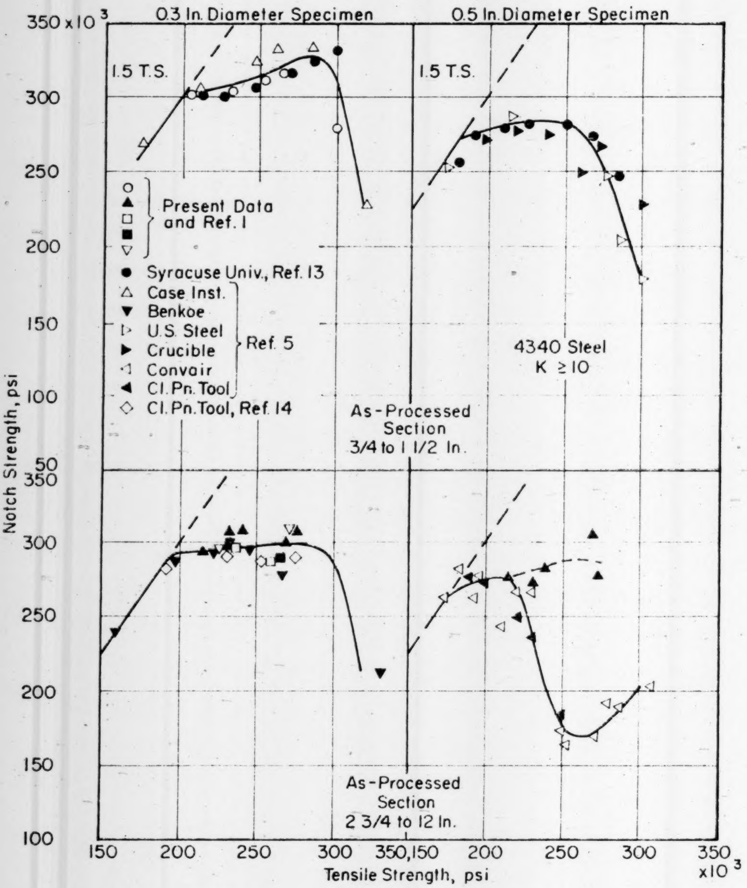
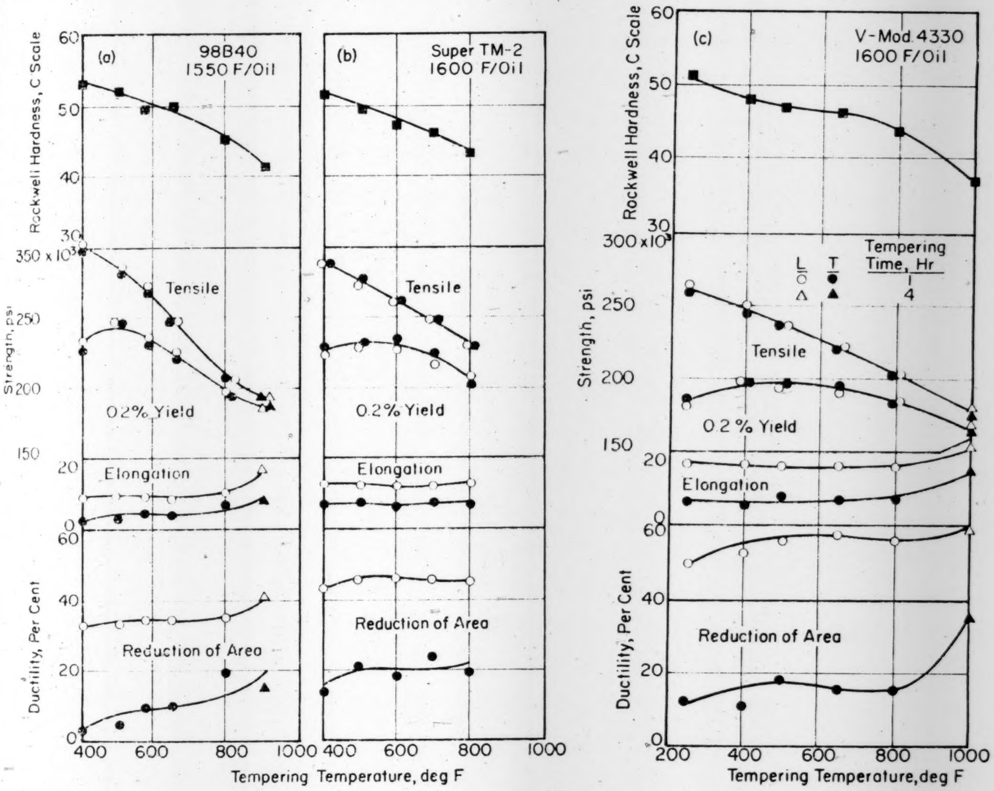


FIG. 10.—Effects of As-Processed and As-Tested Section Size on Notch Strength of Sharply Notched ($K \geq 10$) 4340 Steel Specimens.

The question of size effect and its discussion is controversial. Dependent on the fracture mechanism proposed as governing, it may be projected that for materials, in general, a size effect must in principle obtain. In practice such size effect phenomena are, indeed, very regularly experienced. However, it has been proposed by Klier, Muvdi, and Sachs (**9,10**) that size effect may regularly arise because limiting notch conditions are not regularly obtained. Data recently obtained for fatigue cracked specimens (11) which are entered in Figs. 8 and 9 support this position. In Fig. 9 notch strength data for comparably heat-treated 4340 steel for several notch forms and specimen sizes are presented. If the sharper notches formed in fatigue and by the action of hydrogen led to greater restraint this should appear in the test data as reduced minimum and augmented maximum notch strengths. These trends are seen to obtain. Thus the most severe notch action is seen to arise in the smallest specimen subject to a hydrogen notch.



(*a*) 98B40 steel. (*b*) Super TM-2 steel. (*c*) 4330 V-modified steel.

FIG. 11.—Hardness, Strength, and Ductility of Group A Alloy Steels as Function of Tempering Temperature.

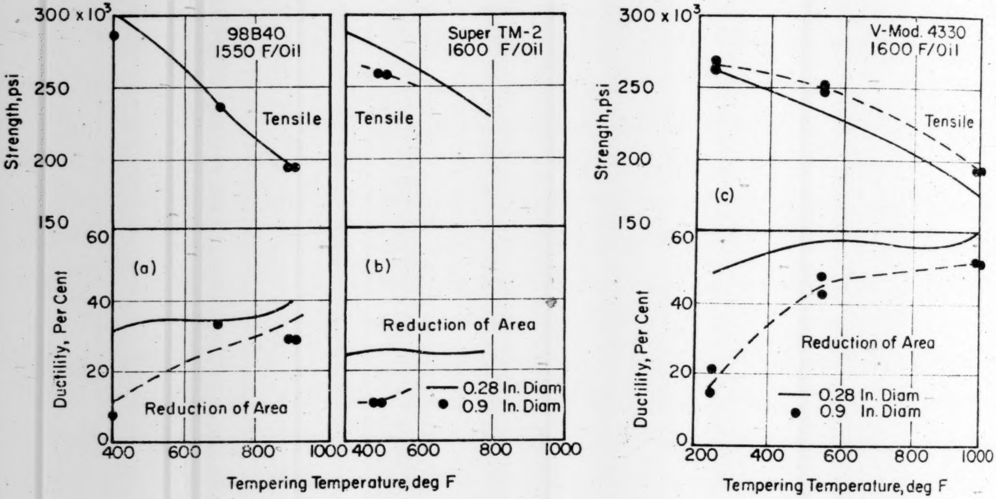
Effect of As-Processed Section Size on Notch Strength:

No attempt was made in the present work to investigate the effects associated with changes in the dimensions of the section from which the test specimens are machined. However, data now available in the literature permit a more thorough evaluation of these effects than was possible before.

The results of notch tension tests on 0.3- and 0.5-in. diameter specimens derived from a number of sources are illustrated in Fig. 10. This graph illustrates the dependence of the notch strength of 4340 steel specimens (longitudinal) having a stress concentration $K \geq 10$ on the as-processed section size for both 0.3- and 0.5-in. diameter specimens. It is observed that the notch strength is appreciably lowered as the as-processed section size is changed from small sections ($\frac{3}{4}$ to $1\frac{1}{2}$-in. diameter) to large sections ($2\frac{3}{4}$ to 12-in. diameter). Furthermore, this effect is insignificant at strength levels below 200,000 psi but increases as the strength level increases above 200,000 psi, the largest strength reduction occurring between 250,000 and 300,000 psi. These observations apply equally to both 0.3- and 0.5-in. diameter specimens.

EXPERIMENTAL RESULTS: B. PROPERTIES OF VARIOUS ALLOY STEELS

The six high-strength steels studied in addition to 4340 steel fall into two groups related by chemical composition. The first group (group A), composed of 98B40, 4330 V-modified, and Super TM-2, is closely related to 4340 steel and sufficiently so that properties changes in these steels can be considered as variations in the properties of 4340 steel to which they should be directly compared. The remaining three steels (group B) are all high-silicon steels and all temper as high-silicon steels should—that is, they resist softening over a relatively long temperature range. When the temperature is sufficiently elevated that softening is induced, the reduction of strength with increase in temperature is very rapid, and the strength in this tempering temperature interval is difficult to control. The first group of steels then can conveniently be heat treated to give a range of tensile strengths, while the second group of steels are designed to yield a relatively constant high tensile strength after relatively high-temperature tempering. These latter steels have for the most part been developed with the objective of outperforming 4340 steel at its maximum possible strength or to allow high-temperature baking for the elimination of hydrogen embrittlement. The two groups of steels can best be considered separately.



(*a*) 98B40 steel. (*b*) Super TM-2 steel. (*c*) V-modified 4330 steel.

FIG. 12.—Strength and Ductility of Group A Alloy Steels as Functions of Tempering Temperature for 0.9-in. Diameter Specimens.

Group A Steels:

Tensile Properties—The tensile properties of 4330 V-modified, 98B40, and Super TM-2 alloys are presented in Fig. 11 as measured for the 0.28-in. specimen. For each steel the hardness increases smoothly as the tempering temperature is reduced and the maximum value is obtained at the minimum tempering temperature. There is a parallel change in the tensile strength, while the yield strength first increases with reduction in temperature, passes through a maximum, and then drops. These data compared to the results for 4340 steel are summarized in Fig. 20.

Because of its low carbon content the 4330 V-modified alloy has reduced hardness and strength properties at the lowest temperature tempered conditions. Since the steel possesses a mild secondary hardening characteristic, as the tempering temperature increases the strength characteristics are relatively slowly reduced and become equal to those for 4340 steel at the intermediate and high tempering temperatures.

Super TM-2 also possesses mild secondary hardening characteristics and develops relatively high tensile strength at the maximum tempering temperature. The 98B40 steel studied was of relatively

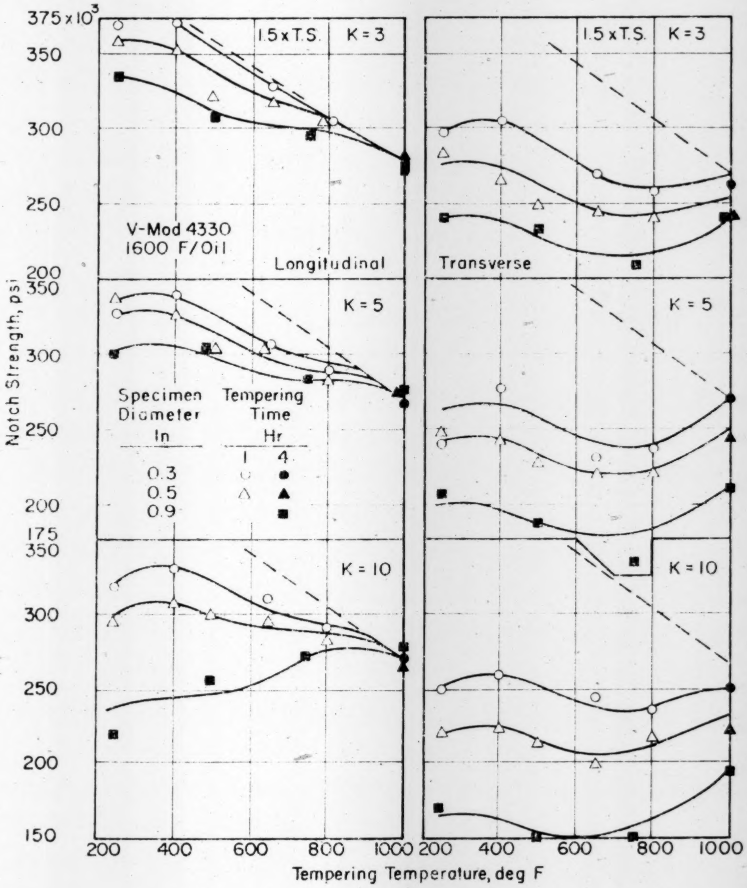


FIG. 13.—Dependence of Notch Strength of 4330 V-Modified Steel on Tempering Temperature and Specimen Diameter.

high carbon content. Consequently it developed the maximum hardness and tensile strength for the low tempering temperatures. This relatively high strength condition was rapidly lost with increasing lated to that for 4340 steel, lay at lower strength levels because of the lower strength of the steel.

The ductility of the steels depended rather obviously on the carbon content

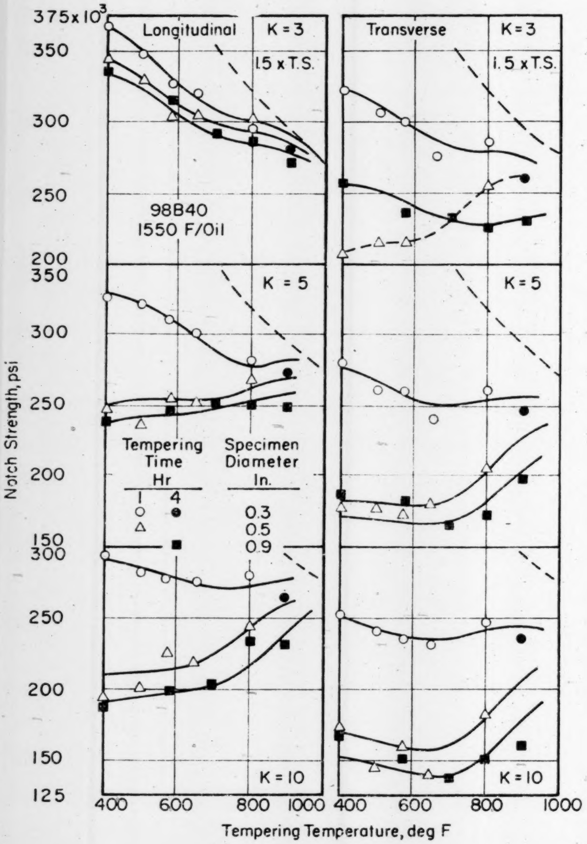


FIG. 14.—Dependence of Notch Strength of 98B40 Steel on Tempering Temperature and Specimen Diameter.

tempering temperature due to the low alloy content of the steel.

The yield strength versus tempering temperature behaviors of Super TM-2, 98B40, and 4340 alloys were nearly identical, being nearly constant in the range 400 to 700 F. For temperatures increasing above 700 F the yield strength dropped smoothly for all steels. The yield strength curve for 4330 V-modified, obviously re-

and orientation of the specimen in the forged section. With increasing carbon content, ductility was first slowly and then abruptly lost. The ductility varied regularly but only slightly as the tempering temperature was varied and tended to maximize at the highest tempering temperatures. The transverse ductility paralleled the longitudinal ductility but for all steels was relatively much reduced. There

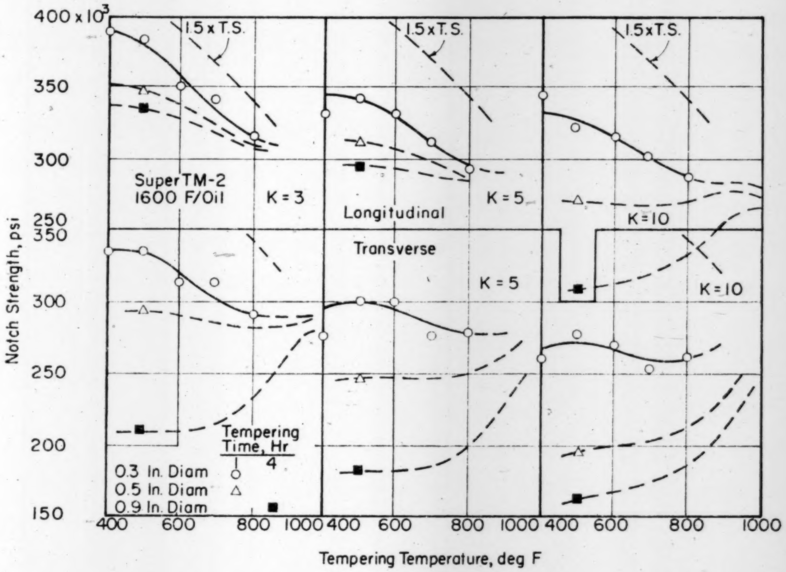


FIG. 15.—Dependence of Notch Strength of Super TM-2 Steel on Tempering Temperature and Specimen Diameter.

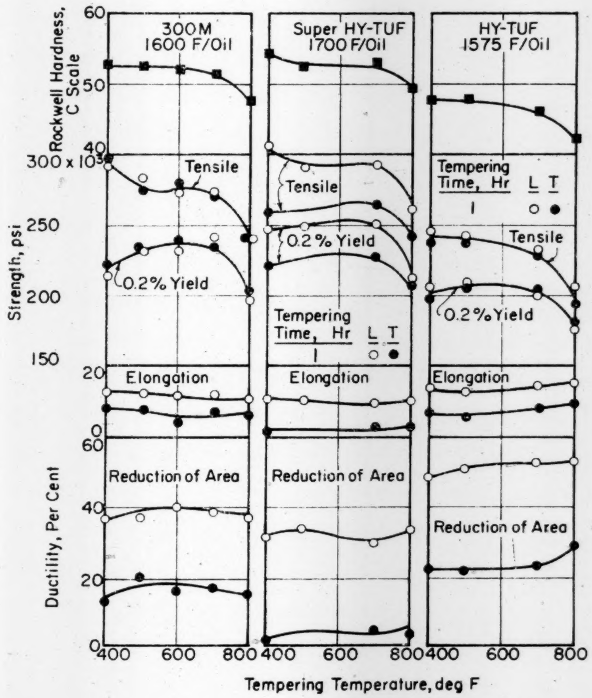


FIG. 16.—Hardness, Strength, and Ductility of 300 M, Super Hy-Tuf, and Hy-Tuf Steels as Functions of Tempering Temperature.

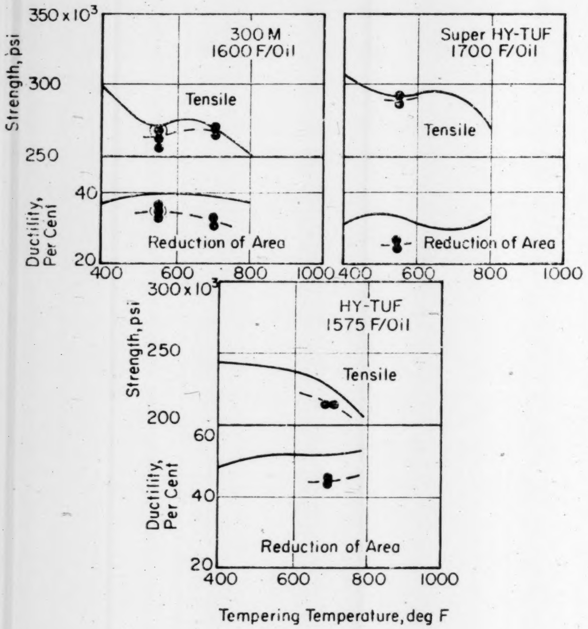


FIG. 17.—Strength and Ductility of 300 M, Super Hy-Tuf, and Hy-Tuf Steels as Functions of Tempering Temperature for 0.9-in. Diameter Specimens.

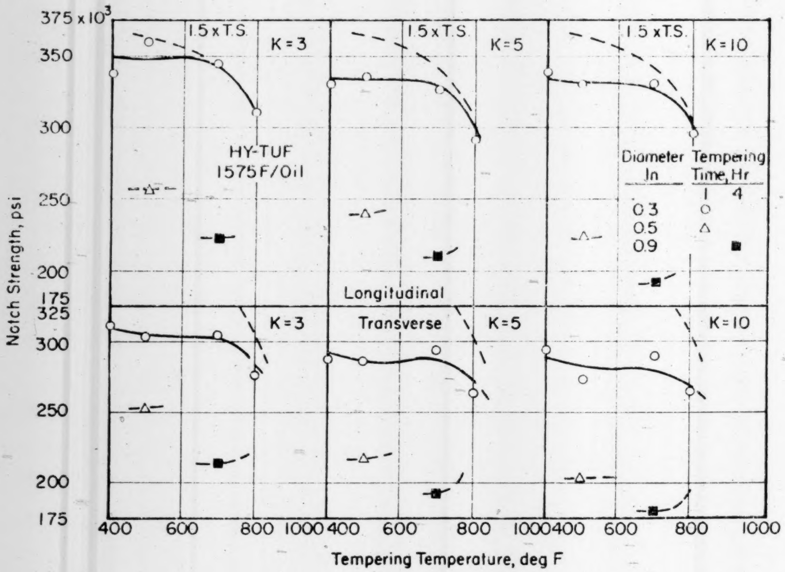


FIG. 18.—Dependence of Notch Strength of Hy-Tuf Steel on Tempering Temperature and Specimen Diameter.

is also indicated some inversion in order with Super TM-2 and 4340 steel possessing the greatest transverse ductility followed by 4330 V-modified and 98B40. This behavior is perhaps due to differences in the forging histories of the different materials and probably should not be given undue weight.

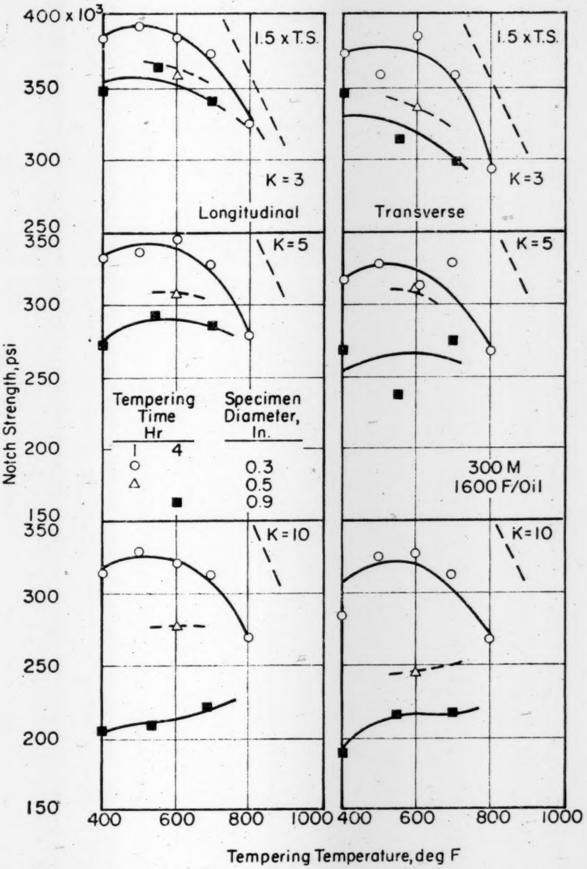


FIG. 19.—Dependence of Notch Strength of 300 M Steel on Tempering Temperature and Specimen Diameter.

Tensile Properties of 0.9-in. Specimens.—The tensile strength and reduction of area in the longitudinal direction for 0.9-in. specimens are given in Fig. 12. In the low-alloy heat-treatable steels it is to be expected that as the quenched section increases in size slack-quenching would be increasingly difficult to suppress. This would lead to reduced strength. Of the three steels, 98B40 yields the same tensile strength in the two specimen sizes, Super TM-2 gives reduced tensile strength, while 4330 V-modified gives increased tensile strength. All three steels in the 0.9-in. diameter specimen give reduced ductility. The tensile strength changes are moderate at most but the ductility changes are large.

Notch Properties.—The notch tensile strengths for the steels at the different specimen sizes and notch configurations in the longitudinal and transverse directions are summarized versus tempering temperature in Figs. 13, 14, and 15. In general, increase in strength level, specimen size, and notch acuity leads to reduced notch strength. A change in specimen orientation from longitudinal to transverse severely emphasizes the nocth sensitivity for all steels. The notch strength varies with tempering temperature for the steels here compared, much as does that for 4340 steel (Fig. 21).

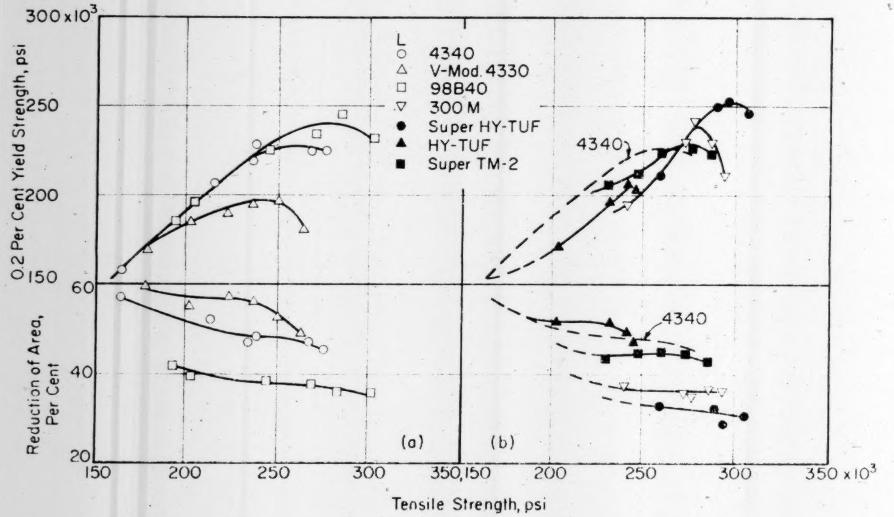


FIG. 20.—Yield Strength and Reduction of Area as Functions of Strength Level for the High-Strength Steels Studied.

Group B Steels:

The tensile properties data for steels Hy-Tuf, Super Hy-Tuf and 300 M are summarized in Fig. 16 for the 0.28-in. specimen. These steels are designed for use after tempering at about 600 F, which develops the maximum yield strength. In the present work the additional tempered conditions were examined to provide a reference framework for use of the steels.

Hy-Tuf is a steel of reduced carbon content and it consequently develops a reduced maximum hardness and tensile strength. The yield strength is nearly constant in the tempering temperature interval 400 to 700 F and equals or slightly exceeds 200,000 psi. The ductility in both the longitudinal and transverse directions is considered good. Super Hy-Tuf and 300 M are steels projected for use at a tensile strength level of 270,000 psi. With a potential yield strength level of 240,000 psi a yield strength to tensile strength ratio of 0.9 is approximated. As the specimen size is increased to 0.9-in. diameter (Fig. 17) the tensile strength is either unaltered or moderately reduced. Again, as for the steels earlier reviewed, the ductility is reduced.

Notch Properties.—The notch strength data for Hy-Tuf steel are presented in Fig. 18. In the small specimens, even for the sharply notched condition, the steel possesses nearly the ideal strength level and this is true for both longitudinal and transverse specimens. As the specimen size is increased, the notch strength values tend to fall.

The notch strength data for 300 M are

presented in Fig. 19. The steel possesses little directionality effect and possesses relatively high notch strength. The notch strength level, however, is reduced by both increasing notch acuity and increase in section.

The notch strength data for Super Hy-Tuf were anomalous with the intermediate 0.5-in. diameter specimens giving the minimum notch strength. This suggests that the most favorable heat treatments for the steel composition had not been developed at the time the testing was done.

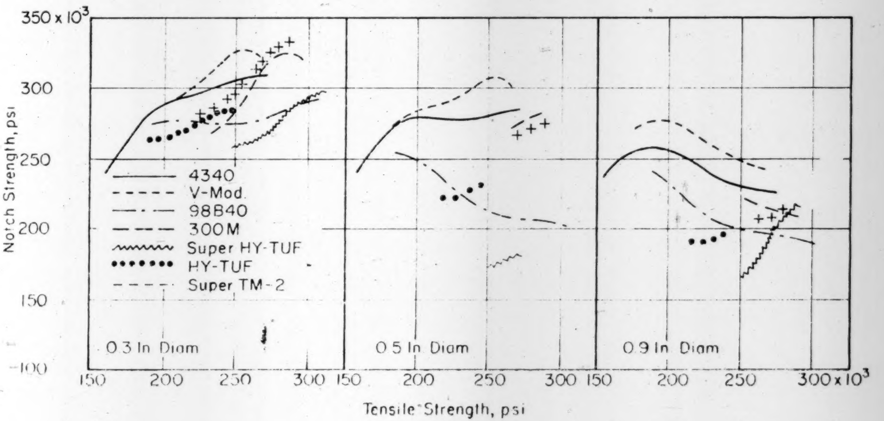


FIG. 21.—Dependence of Notch Strength on Tensile Strength for the High-Strength Steels Studied.

COMPARISON OF THE TENSILE AND NOTCH TENSILE DATA FOR THE STEELS STUDIED

The selection of a high-strength steel may be based on several criteria, and there is no unanimous agreement on which of these criteria, in general, governs. The data for the steels examined are compared in Figs. 20 and 21 for yield strength in the 0.28-in. diameter specimens, for reduction of area, and for notch strength ($K = 10$) with specimen diameter as parameter.

In a general sense it is evident from Fig. 20 that a high yield strength to tensile strength ratio at high strength levels tends to be incompatible with high ductility. Thus those steels which have maximum ductility tend to have minimum yield strength at a given tensile strength level. By this type of comparison it is evident that 4340 steel is a competitive steel for high-strength applications.

The notch strength data tend to evaluate the respective steels in the same manner as the ductility, but this ordering is not exact. It is again seen that the 4340 steel is a steel with excellent competitive properties as determined in the notch tests. However, it is desirable to emphasize an important fact at this time. The high-strength proprietary steels have been designed to attain strength levels that cannot be reached with 4340 steel, in general. If these very high strengths are deemed necessary, prudence must dictate the selection of the best steel for the intended application; the data in Figs. 20 and 21 can be of assistance in this selection. For all steels of the quench-and-temper type which may be used at very high strength levels, notch sensitivity and associated intensification of notch sensitivity through the action of size effect are factors of major concern.

SUMMARY AND CONCLUSIONS

The tensile and notch tensile properties of seven low-alloy quenched-and-tempered steels have been determined variously for specimens to 0.9-in. diameter. The following summary of conclusions is offered:

1. The tensile and yield strengths for the steels studied do not seriously differ with specimen size change, even to the largest sizes and highest strength levels for which data are available, except when residual stresses or slack-quenched structures or both are present.

2. Those indexes of ductility in the tension test—namely, elongation and reduction of area—however, are reduced with specimen size increase. Ductile-brittle transitions for these quantities are indicated although these transitions have not been directly measured.

3. Notch strengths for high-strength conditions for all the steels studied are emphatically reduced as the section size is allowed to increase.

4. The changes in tensile and notch tensile properties are numerically altered by the additional factors of specimen orientation, chemical composition, and stress concentration, while the qualitative trends parallel those measured for 4340 steel.

Acknowledgments:

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MECHANICAL PROPERTIES OF A CHROMIUM - 1 PER CENT COLUMBIUM ALLOY

By Raymond E. Cairns, Jr.[1] and Nicholas J. Grant[2]

Synopsis

A number of properties were determined for an extruded, arc-melted chromium-base alloy containing 1 per cent columbium and scavenged with 1 per cent yttrium in the recrystallized and hot-worked conditions. The brittle-to-ductile transition temperature varied between 325 and 575 F depending upon the metallurgical history. The dynamic modulus of elasticity, measured between room temperature and 2000 F, varied from 41 to 32 million psi. The oxidation resistance of the alloy was comparable to Nichrome V at 1800 F but was somewhat inferior at 2000 F. Stress rupture properties were determined at 1500 to 2000 F; high strength and good ductility were noted.

During the past decade the demand for materials to withstand higher service temperatures has become increasingly important. Nickel and cobalt alloys are generally restricted to use below about 2000 F for strength reasons; the refractory metals are restricted in use because of exceedingly poor oxidation resistance.

Chromium has long been of interest as a base metal for alloys to be used at temperatures above 1800 F, with some indication that 2400 or 2500 F might be attainable. Chromium possesses a relatively high melting temperature (3450 F), a high elastic modulus (41×10^6 psi), high heat conductivity, a low coefficient of expansion, a relatively low specific gravity, and good oxidation resistance. Chromium, however, like several other body-centered cubic metals, suffers from a relatively high transition temperature for ductile to brittle fracture, which has hindered its engineering usefulness for the time being.

Accumulated evidence from studies conducted in our laboratory and elsewhere (**1**),[3] indicates that certain chromium alloys possess high strength properties at 1800 to 2200 F. Unfortunately, the few alloys that have been reported have been tested in a fairly limited way (**1**, **2**). In particular, additions of tungsten, molybdenum, tantalum, columbium, and titanium to chromium have resulted in significant improvements in high-temperature properties. Because additions of at least 2 per cent of these elements raise the brittle-fracture transition temperature of chromium

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper.

(3) to very high values, only small ingots of several pounds total weight have been made to avoid severe ingot cracking. Therefore, to obtain an extrudable alloy and to produce an adequate supply of bar stock for testing, the chosen composition was restricted to 1 per cent columbium; the solubility limit of columbium in chromium is about 2 weight per cent. Based on data regarding oxidation resistance (4, 5) and beneficial effects of strong scavenging elements on hot working and ductility, an addition of 1 per cent yttrium was planned.

This investigation includes: (1) determination of the ductile to brittle transition temperature for two conditions of the alloy; (2) determination of the dynamic modulus of elasticity from room temperature to 1800 F; (3) measurements of oxidation resistance at 1800 and 2000 F; and (4) stress-rupture properties at 1500, 1800, and 2000 F.

EXPERIMENTAL PROCEDURE

Alloy Preparation:

The chromium was hydrogen-reduced electrolytic grade, the composition of which is shown in Table I. Nitrogen at a lower level was desired, but with the yttrium addition the nitrogen was not considered to be detrimental. The columbium and yttrium analyses, to the extent reported by the suppliers, are also shown in Table I.

The alloy was made by nonconsumable arc melting (cold copper crucible) in a purified helium atmosphere (final step over 1600 F titanium chips) at 25 mm pressure. The ingot was crushed and remelted twice to assure maximum homogeneity, and then cooled in the helium atmosphere. The final dimensions of the ingot were 3 in. diameter by 8.5 in. long.

TABLE I.—PURITY OF MELTING MATERIALS IN WEIGHT PER CENT.

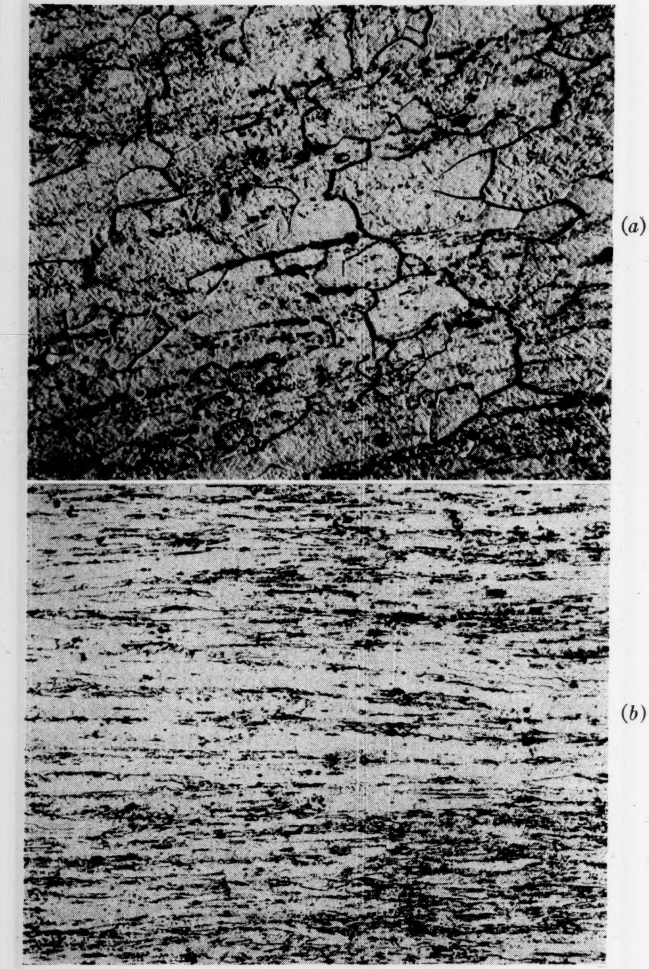
Chromium		Columbium		Yttrium	
Impurity	Amount	Impurity	Amount	Impurity	Amount
Carbon	0.008	Iron	0.01	Calcium	1.0
Sulfur	0.008	Silicon	0.01	Yttrium	98.2
Silicon	0.02	Titanium	0.01		
Iron	0.02	Tantalum	0.15		
Oxygen	0.015	Boron	0.0001		
Hydrogen	0.0005	Nitrogen	0.03		
Nitrogen	0.010	Oxygen	0.07		
		Carbon	0.01		

TABLE II.—ANALYSIS OF EXTRUDED BAR STOCK.

Element	Per Cent
Oxygen	0.011
Nitrogen	0.013
Columbium	0.90
Yttrium	0.24

Large losses of yttrium were anticipated; hence the 1 per cent addition to the original melt. Table II gives the analysis of the extruded bar stock. The oxygen and nitrogen contents, in view of the 0.24 per cent yttrium content, were considered to be satisfactory for hot workability.

Conversion to bar stock was by means of hot extrusion. The ingot was cleaned, canned in an evacuated mild steel container (final diameter, $3\frac{5}{8}$ in.), heated to 2350 F, soaked for 2 hr, rapidly transferred to the press, and extruded. The extrusion die, 1.25-in. bore, was preheated to 900 F. The ram speed was 145 in. per min; the average pressure was 675 tons; and the extrusion ratio (reduction of area) was 8.5:1. The resultant bar, obtained after an initial failure to extrude, measured 1.25 in. diameter by 72 in. long, and it is believed to be the largest chromium alloy extrusion reported to date. The hot bar was cooled slowly in vermiculite.



(a) After recrystallization treatment at 2100 F for 1 hr.
(b) In warm-worked condition (1900 F extrusion).

Fig. 1.—Prior Processing Conditions of Chromium Alloy Containing 1 per cent Columbium; Electrolytic Oxalic Etch (× 250).

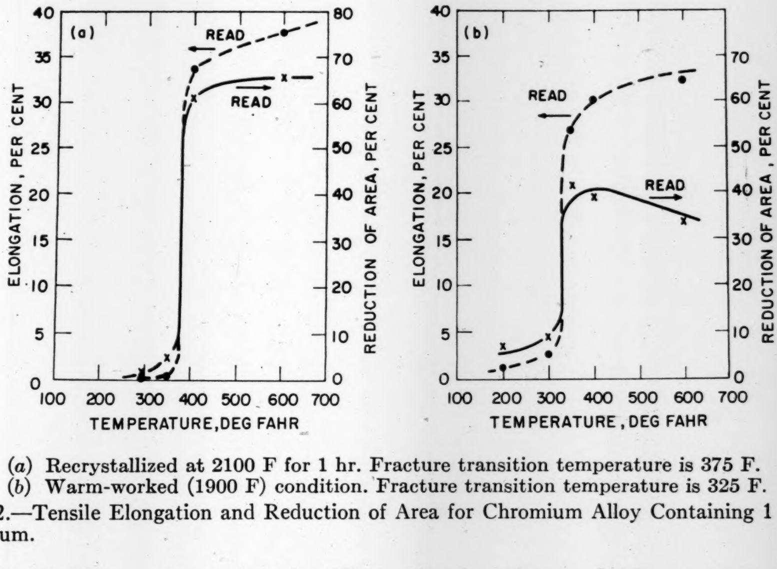
Except for a few minute surface cracks, the bar was completely sound. To produce more bar stock, the long bar was cut into 4-in. lengths and was recanned in austenitic stainless steel. Most of the pieces were heated to 2200 F and extruded with a reduction ratio of about 4:1; several lengths were extruded at 1900 F.

Alloy Condition:

The alloy was investigated for two prior-processing conditions:

1. *As-recrystallized.*—Recrystallization was accomplished by heating the extruded alloy slowly over several hours to 2100 F. It was held for 1 hr, transferred to a 1700-F furnace, and furnace cooled to 1200 F, from which it was air cooled to room temperature. Figure 1(*a*) hows that the structure is fully recrystallized but not equiaxed. The ASTM grain size is 3 to 6. The excess phase particles are a mixture of yttrium oxide, yttrium nitride, and the intermetallic Cr_2Y.

2. *Warm-worked.*—Instead of extruding at 2200 F, as was the situation for the material that was recrystallized, several short lengths were extruded at 1900 F. Figure 1(*b*) shows that the structure is coarsely fibrous and in the warm-worked condition. As a result of the lower extrusion temperature the alloy is harder by 9 Rockwell B points, equivalent to about 10 per cent reduction by cold work at room temperature.



(*a*) Recrystallized at 2100 F for 1 hr. Fracture transition temperature is 375 F.
(*b*) Warm-worked (1900 F) condition. Fracture transition temperature is 325 F.

FIG. 2.—Tensile Elongation and Reduction of Area for Chromium Alloy Containing 1 per cent Columbium.

Mechanical Testing:

Tension tests were performed in air at a strain rate of 0.02 in. per in. per min using button head specimens with a liberal fillet at the shoulder. The specimen dimensions of the reduced section were 0.160 in. diameter by 1.25 in. long. All specimens were carefully electropolished in a 95 per cent glacial acetic acid containing 5 per cent perchloric acid solution, using an open-circuit voltage of 25 v, current density of 12 amp per sq in., and a polishing time of about 2 min. In temperature tests all specimens were held at temperature for 30 min prior to testing.

The dynamic modulus of elasticity was determined from room temperature to 1800 F on an Elastomat apparatus. The test specimens, accurately machined to 0.500 in. diameter by 6 in. long, were made from bar stock that had been fully recrystallized at 2100 F for 1 hr. The temperature was carefully controlled to ±3 F. The Elastomat measures the fundamental frequency in transverse vibration with the specimens as a free-

free beam. The specimen is excited electromechanically by means of an attached fine wire from the frequency source. The pertinent equation for the dynamic modulus is:

$$E = 9.18 \times 10^{-6} \cdot \frac{l^4}{d^4} \cdot \frac{W}{l(1 + \alpha T)} \cdot f^2$$

where:

l = length,
d = diameter,
w = weight,
α = coefficient of expansion (5.4×10^{-6} per deg Fahr),
T = temperature, and
f = fundamental frequency.

Stress rupture tests were performed in air on the same type of specimen as was used for the tension tests. Temperatures were controlled to ±5 F over the 1.25 in. gage length.

In spite of the successful extrusion of the ingot and re-extrusion of the large diameter bar, adequate material was unavailable to extend the test program to higher temperatures, for longer times, or for additional prior histories for the alloy.

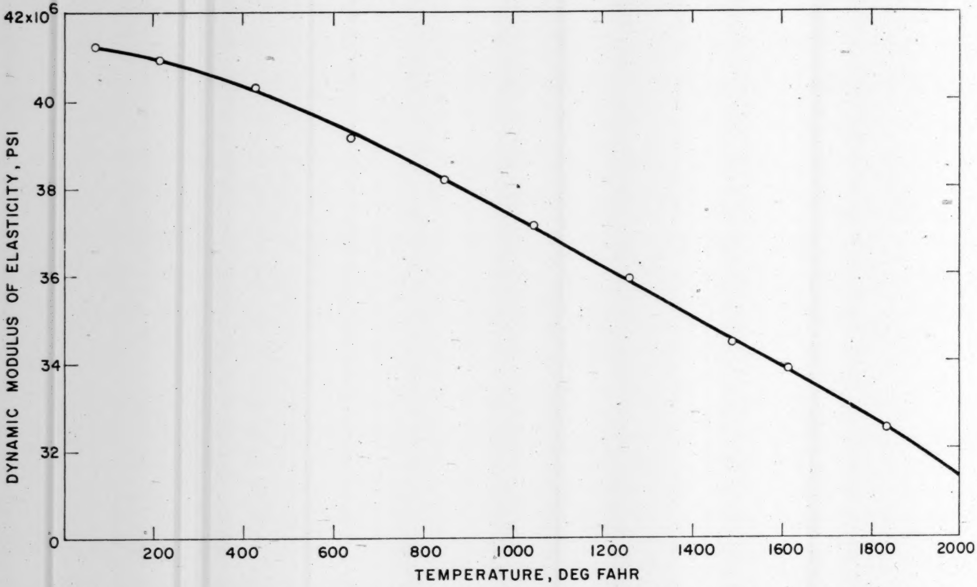


FIG. 3.—Dynamic Elastic Modulus Versus Temperature for Recrystallized Chromium Alloy Containing 1 per cent Columbium.

Oxidation Resistance:

Tests were carried out at 1800 and 2000 F, using square-based rectangular prisms having edge dimensions of 0.375 by 0.750 in. high. Specimens were surface ground, briefly electropolished, and cleaned. The specimens were contained in platinum boats to prevent loss of spalled oxide. Although the measurements made were accurate to about 1 mg (weight gain), evaporation losses of chromium were not accountable. Natural convection of air prevailed by keeping both ends of the furnace tube open. For comparison purposes a Nichrome V alloy (80-20 nickel-chromium) was included in the tests. Temperatures were controlled to about

±7 F. Oxide penetration measurements were made by means of microexamination and microhardness tests (Knoop, with 100-g load).

EXPERIMENTAL RESULTS AND DISCUSSION

Fracture Transition Temperature:

Figure 2(*a*) is a plot of ductility in a tension test versus temperature for the recrystallized alloy. The resultant transition temperature of 375 F is measured by the intercept of the steep portion of the curve on the temperature axis.

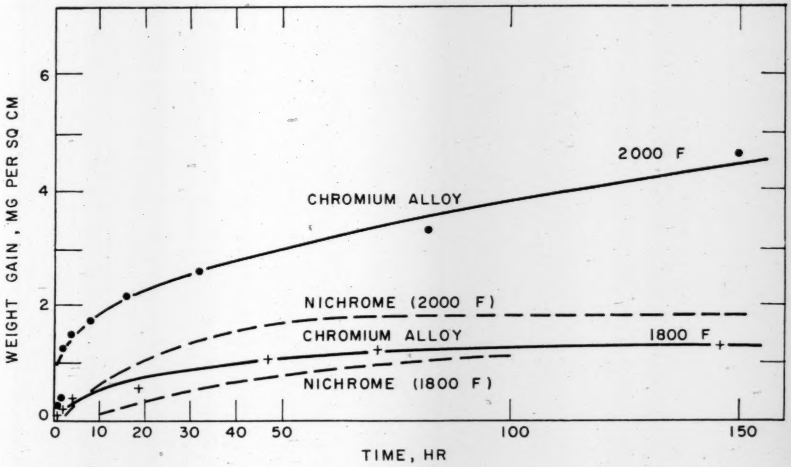


FIG. 4.—Weight Gain Versus Time Data for Oxidation of Chromium Alloy Containing 1 per cent Columbium Tested in Air at 1800 and 2000 F; Nichrome V Shown for Comparison.

Figure 2(*b*) is a similar plot for the warm-worked structure in which the transition temperature decreased to 325 F. It will be noted that Fig. 2(*b*) shows an enlarged "tail" at the low test temperatures. If one chose to use a low value of reduction of area, for example, 5 per cent, as a measure of ductile behavior, the warm-worked condition would have a transition temperature of about 300 F. Even for reduction of area values as high as 20 per cent, there is no necking of the specimen; one must be at a temperature just above the upper break in the curve to achieve true ductility. This behavior is characteristic of chromium.

The lower-temperature tension test data are shown in Table III. The highest values are shown at 400 F for the recrystallized alloy (72,800 psi) and at 350 F for the warm-worked alloy (89,700 psi), both values being found in the fully ductile (necked) region.

Modulus of Elasticity:

Figure 3 shows the variation of elastic modulus with temperature. The curve is nearly linear from about 600 to 1800 F. In comparing the lower temperature values with those of unalloyed chromium, it is noted that the chromium - 1 per cent columbium alloy does not exhibit the sharp discontinuity shown by pure chromium at about 95 F. Pursey (6) showed that small amounts of impurities or alloying elements lower the temperature at which this discontinuity occurs. The chromium - 1 per cent columbium alloy has a room-temperature modulus of 41.3 × 10^6 psi and has about the same elastic modulus at 2000 F that steel has at room temperature, offering an attractive design parameter.

Oxidation Studies:

The results of air oxidation tests have been reported for chromium and some of its alloys by several investigators (**4, 5, 7**). The results of the current study of the chromium - 1 per cent columbium alloy are shown in Fig. 4, along with comparative data on the 80-20 nickel-chromium alloy. At 1800 and 2000 F the oxide is protective; the results at 1800 F are more nearly parabolic but show a higher rate at 2000 F. Because of volatilization losses of chromium at 2000 F, it is impossible to say much about the actual rate of oxide formation. At 1800 F the chromium - 1 per cent columbium alloy shows an oxidation rate nearly equal to that of Nichrome V; however, at 2000 F the rate is significantly worse. In 100 h at 2000 F, the chromium alloy weigh gain is about 2.5 times that of Nichrome V.

At 1800 F, no spalling was noted; however, at 2000 F, considerable spalling took place, accounting in part for the steeper curve in Fig. 4.

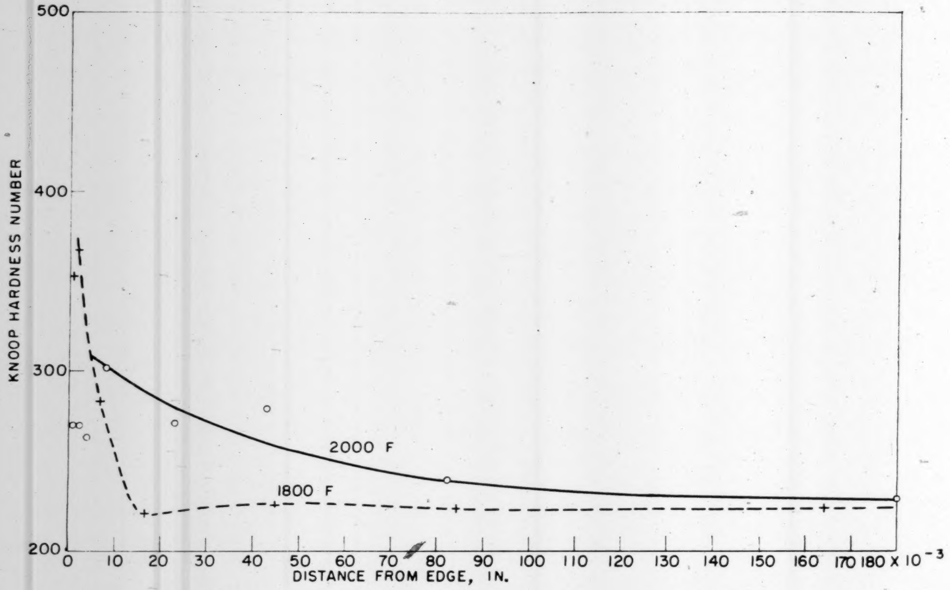


Fig. 5.—Change of Hardness with Depth of Penetration of Oxygen and Nitrogen During 150 Hr of Exposure to Air at 1800 and 2000 F for Chromium-Columbium Alloy.

Figure 5 is a plot of Knoop hardness versus distance from the original surface for the chromium-columbium alloy after 150 hr exposure to air at 1800 and 2000 F. There is an increase in hardness to a depth of 0.010 to 0.015 in. at 1800 F, with a fairly steep hardness gradient at the surface. This hardness increase is due to some internal oxidation and to nitrogen solution. At 2000 F, the average penetration is greater, but the hardness gradient is less severe, presumably because diffusion of oxygen and nitrogen takes place more rapidly. Penetration to a depth of possibly 0.050 in. is indicated;

which was confirmed by metallographic observations. Incidentally, grain growth did not occur during these exposure tests.

TABLE III.—LOW-TEMPERATURE TENSILE DATA.

Condition	Test Temperature, deg Fahr	Ultimate Tensile Strength, psi	Per Cent Elongation	Reduction of Area, per cent	Transition Temperature, deg Fahr
Recrystallized	300	42 700	0.8	1.0	375
	350	42 200	2.5	1.0	
	400	72 800	30.8	67.8	
	600	61 700	32.5	75.7	
Warm Worked	200	83 500	3.4	2.5	325
	300	85 500	4.2	5.5	
	350	89 700	20.7	53.8	
	400	85 500	17.7	60.7	
	600	80 400	17.0	64.7	
Recrystallized, then oxidized for 100 hr at 1800 F	400	25 800	0.6	0	570
	500	51 300	1.3	0	
	550	52 200	0	0	
	600	71 700	28.0	69.2	

TABLE IV.—STRESS RUPTURE DATA FOR CHROMIUM ALLOY CONTAINING 1 PER CENT COLUMBIUM.

Condition	Test Temperature, deg Fahr	Stress, psi	Rupture Life, hr	Elongation, per cent	Reduction of Area, per cent
Recrystallized	1500	35 000	153.0	10.9	[a]
		40 000	22.1	16.1	[a]
		50 000	1.2	17.3	[a]
	1800	15 000	232.9	34.4	36.8
		20 000	32.4	21.3	41.8
		22 000	12.0	18.3	32.9
		25 000	10.5	20.4	32.2
		30 000	3.9	13.8	25.4
	2000	9 000	144.2	32.7	52.8
		15 000	2.9	8.3	17.9
		25 000	0.3	21.6	36.9
Warm worked	1500	55 000	11.8	4.5	15.8
		40 000	264.5	8.7	[a]
	1800	17 500	380.3	29.4	40.0
		20 000	38.5	18.6	35.2
		25 000	17.3	27.9	53.8
		30 000	5.2	27.8	64.0

[a] Specimen broke at shoulder.

A series of machined and polished tension test specimens, exposed for 100 hr at 1800 F (originally in the 2100 F recrystallized condition, equivalent to a fracture transition temperature of 375 F), was tested in tension to determine the change in fracture behavior as a result of atmospheric contamination. Table III shows the tension test data; the new transition temperature is about 570 F, an increase of about 200 F over the recrystallized condition.

Stress-Rupture Properties:

The stress-rupture test results are listed in Table IV and plotted in Fig. 6.

At 1500 and 1800 F, the warm-worked structure is somewhat stronger than the recrystallized structure. No comparison was made at 2000 F due to a shortage of test specimens. In particular, note that at 2000 F the curve breaks slightly upward, suggesting that internal oxidation or nitrogen solution, or both, takes place in the longer time tests, leading to strengthening of the structure.

At 1500 F, all tests failed with a transcrystalline fracture. At 1800 F the mode of fracture changed to intercrystalline and prevailed in tests at 2000 F.

Figure 7 compares the stress for

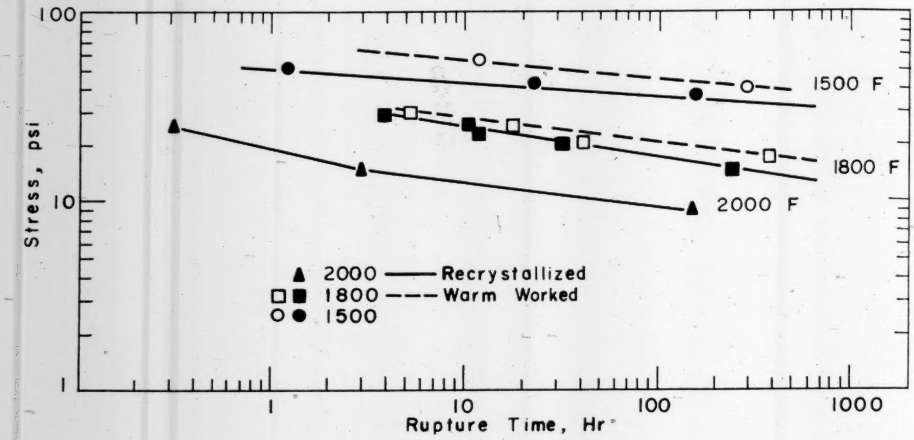


FIG. 6.—Log Stress Versus Log Rupture Time for Recrystallized and Warm-Worked Chromium Alloy Containing 1 per cent Columbium at 1500 to 2000 F.

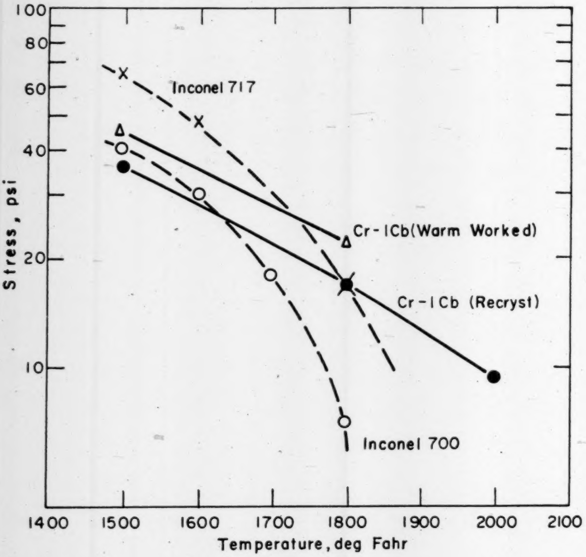


FIG. 7.—Stress for 100-Hr Rupture Life Versus Test Temperature for Chromium Alloy Containing 1 per cent Columbium, Compared to Wrought Inconel 700 and Cast Inconel 717.

100 hr rupture life (interpolated from Fig. 6) versus temperature for the chromium - 1 per cent columbium alloy with values reported in the literature for Inconel 700, one of the best wrought nickel-base alloys and Inconel 717, one of the best cast nickel-base alloys. The strength and ductility performance of the chromium alloy is good, and the properties are interesting for use above 1500 F. Note in Table IV that ductility values at these temperatures are quite adequate.

SUMMARY AND CONCLUSIONS

An extruded chromium - 1 per cent columbium alloy, treated with yttrium, was tested to establish tension properties, transition temperature for ductile-to-brittle fracture, oxidation resistance, and stress-rupture properties from 1500 to 2000 F. The following observations are made:

1. The transition temperature of the recrystallized alloy was 375 F in tension tests. In the warm-worked state, it fell to 325 F. Exposure at 1800 F for 100 hr increased the transition temperature of the recrystallized material by about 200 to 570 F, probably due to nitrogen contamination.

2. Oxidation resistance at 1800 F is almost as good as that of 80-20 nickel-chromium alloy in terms of weight gain, but solution of nitrogen and oxygen is damaging in a 100-hr exposure test. At 2000 F, contamination proceeds more rapidly, partially due to spalling of the oxide.

3. Elastic modulus falls from about 41 × 10^6 psi at room temperature to about 31.5 × 10^6 psi at 2000 F.

4. Creep-rupture properties are very good for such a dilute alloy in the temperature range 1500 to 2000 F. At 1800 to 2000 F, this simple, solid-solution alloy shows stress rupture values which compare favorably with those of the best wrought, age hardened, nickel-base alloys. The warm-worked condition leads to better creep-rupture strength at 1500 to 1800 F than is shown by the recrystallized structure.

Acknowledgment:

The authors express their appreciation to the Department of the Navy, Bureau of Naval Weapons, for support of this program.

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DISCUSSION

MR. C. T. SIMS[1] (*presented in written form*).—The authors are to be commended for their continued interest in chromium systems. Despite the drawbacks which appear to plague chromium, studies such as this make positive progress toward chromium-base, oxidation-resistant alloys for service above 2000 F.

One of the most important parts of this particular work, which should be emphasized, is that a reasonably workable, machinable, handleable chromium-base alloy of a practical nature was produced. This was done using only "commercial" hydrogen-reduced electrolytic grade chromium rather than expensive high-purity material, such as chromium produced by the iodide process. The tensile ductile-to-brittle transitions measured for the alloy using this material are similar to those found for tungsten and tungsten-base alloys.

The authors show no microstructures of the oxidation-nitrogenation front and attribute the hardening found to internal oxidation and nitrogen solution. In fairly similar chromium-base alloys, also containing columbium, a dispersed phase, possibly Cr_2N or CbN, has been seen near the surface.[2] Have the authors checked for a possible dispersion of Cr_2N? Further additions of columbium, up to 5 per cent, will create a continuous nitride layer of much greater hardness,[2] and will also seriously destroy the good oxidation resistance demonstrated.

The strength characteristics of this system should be reemphasized. Perhaps of more importance than the specific rupture properties reported is the lower stress-temperature slope shown compared to the nickel-base alloys. Also, the strength improvement shown may be due to more than solid solution alone. There appears to be about 0.03 per cent total interstitials present, which should provide some fairly effective dispersion hardening as, perhaps, chromium or columbium carbides modified with oxygen and nitrogen. (Solubility of these elements in chromium is estimated at 0.1 to 1.0×10^{-4} per cent at room temperature.[3]) The microstructure shown (Fig. 1 of the paper) and the solutioning effect of warm working on rupture properties (Table IV of the paper) would support this. It further suggests that carbide dispersions should be very effective rupture-strengthening additions to chromium. In fact, it is difficult to understand why strengthening by carbide dispersions, so vital to chromium's sister element molybdenum, has not been investigated for chromium.

MR. NICHOLAS J. GRANT (*author's closure by letter*).—We very much appreciate Mr. Sims' discussion and his emphasis on a number of points. We did not, unfortunately, make positive identification of the phases formed through reaction with the atmosphere but would suspect, also, chromium and columbium nitrides.

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[1] Metallurgy Unit, Materials and Processes Laboratory, General Electric Co., Schenectady, N. Y.

[2] C. T. Sims, unpublished work.

[3] G. T. Hahn, A. Gilbert, and R. I. Jaffee, "The Effects of Solutes on the Ductile-to-Brittle Transition in Refractory Metals," AIME Refractory Metals Symposium, Chicago, Ill., 1962.

The speculation that some strengthening is due to dispersions associated with interstitials is reasonable; however, lower and higher columbium content alloys suggest that the contribution of interstitials is not too great. Carbide strengthening warrants attention, but a higher carbon content does make the casting of larger ingots considerably more difficult.

EFFECTS OF IMPURITY ELEMENTS AND COLD ROLLING ON THE MECHANICAL PROPERTIES OF TITANIUM-5Al-2.5Sn ALLOY AT ROOM AND CRYOGENIC TEMPERATURES

By J. L. Christian,[1] A. Hurlich,[2] J. E. Chafey,[1] and J. F. Watson[3]

Synopsis

The mechanical properties of 19 heats of titanium-5Al-2.5Sn alloy have been determined from 78 to −423 F. Tensile and fusion weld tensile data were determined as well as notched tensile data, which were used as criteria for evaluating the toughness or resistance to brittle failure. The effects of chemical composition, in particular the oxygen and iron contents, and the effects of cold rolling on the strength and toughness of the Ti-5Al-2.5Sn alloy were determined at 78 and −423 F. From these data a specification for low-impurity Ti-5Al-2.5Sn alloy was prepared. Several heats of material produced to this specification were evaluated and were found to be satisfactory for structural applications to liquid hydrogen temperature (−423 F).

A number of investigations have been made on the low-temperature mechanical properties of titanium-5Al-2.5Sn alloy (**1–7**).[4] This investigation supports and adds to the previously published data by providing information on a modification of the alloy which is shown to possess improved toughness. The purpose of the program reported was to determine the suitability of the Ti-5Al-2.5Sn alloy for structural use at cryogenic temperatures.

The interest in titanium for pressure vessel use in missiles and space vehicles is primarily due to its high strength-to-density ratio, particularly at cryogenic temperatures. For example, the Ti-5Al-2.5Sn alloy possesses a 30 to 50 per cent higher strength-to-density ratio at −423 F than high-strength aluminum alloys or cold rolled 300 series stainless steels (**8**). Therefore a number of titanium alloys were evaluated for strength and toughness properties from 78 to −423 F, and the results indicated that the Ti-5Al-2.5Sn alloy possessed the best combination of strength, toughness, and fabricability for structural use at −423 F (**9**). It was noted, however, that large differences in properties, particularly toughness, were obtained from one heat to another in the Ti-5Al-2.5Sn alloy at −423 F. As a result, a more detailed investigation was made.

The first phase of the investigation included the determination of tensile, notched tensile, and fusion weld tensile properties of seven heats of normal commercial (Specification AMS 4910) material. From these data, as well as

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[4] The boldface numbers in parentheses refer to the list of references appended to this paper.

TABLE 1.—CONDITION AND CHEMICAL ANALYSES OF HEATS OF Ti-5Al-2.5Sn ALLOY TESTED.

Heat	Thickness, in.	Temper	Superficial Rockwell Hardness, 15N Scale	Supplier	Composition, weight per cent						
					Aluminum	Tin	Iron	Carbon	Hydrogen	Nitrogen	Oxygen
M-8465	0.040	mill annealed	78	TMCA[a]	5.8	2.4	0.09	0.017	0.0108	0.010	0.103
M-9048	0.027	mill annealed	78	TMCA	5.4	2.5	0.21	0.021	0.015	0.010	0.17
31387	0.020	mill annealed	78	Reactive Metals, Inc.	5.6	2.6	0.24	0.03	0.010	0.015	0.12
D-2073	0.063	mill annealed	80	Crucible Steel Corp.	4.9	2.4	...	0.03	0.0061	0.03	0.16
D-2053	0.016	mill annealed	81	TMCA	5.3	...	0.37	0.026	0.028	0.017	...
M-8394	0.032	mill annealed	78	TMCA	5.6	2.2	0.04	0.015	0.013	0.009	0.17
M-8613	0.040	mill annealed	80	TMCA	5.14	2.53	0.18	0.038	0.0084	0.023	0.167
D-1311	0.020 to 0.036	mill annealed	79	TMCA	5.2	2.5	0.27	...	...	...	0.20
V-1866	0.020 to 0.035	mill annealed	78	TMCA	5.3	2.5	0.50	...	...	...	0.08
V-1867	0.020 to 0.041	mill annealed	77	TMCA	5.2	2.6	0.15	...	...	...	0.07
V-1699	0.041	mill annealed	75	TMCA	5.1	2.8	0.07	0.03	0.014	0.01	0.09
V-1668	0.038	mill annealed	77	TMCA	5.1	2.6	0.10	0.03	0.009	0.02	0.11
V-1669	0.041	mill annealed	78	TMCA	5.0	2.5	0.07	0.03	0.009	0.02	0.15
V-1670	0.040	mill annealed	78	TMCA	5.0	2.6	0.02	0.03	0.011	0.02	0.17
V-1671	0.043	mill annealed	80	TMCA	4.9	2.5	0.07	0.04	0.010	0.02	0.24
M-9048	0.035	12 per cent cold rolled	80	TMCA	5.4	2.5	0.21	0.021	0.015	0.010	0.17
M-8465	0.024 to 0.032	20 to 40 per cent cold rolled	80 to 83	TMCA	5.8	2.4	0.09	0.017	0.0108	0.010	0.103
XT-60080	0.034	mill annealed to 50 per cent cold rolled	76 to 80	Republic Steel Corp.	5.17	2.55	0.19	0.032	0.0012	0.008	0.07
3930131	0.014	mill annealed, vacuum annealed, and stretch formed	78	Republic Steel Corp.	5.45	2.50	0.11	0.035	0.0099	0.011	0.12
3960328	0.017 to 0.125	mill annealed and stretch formed	76 to 78	Republic Steel Corp.	5.28	2.56	0.13	0.028	0.0050	0.010	0.10
3930498	0.010 to 0.015	mill annealed	77	Republic Steel Corp.	5.38	2.53	...	0.04	0.0048	0.007	0.12

[a] Titanium Metals Corp. of America.

from results obtained by other investigators (**2,3,6,7**), it was evident that the amount of interstitial elements (carbon, oxygen, nitrogen, hydrogen) present in the material strongly affected the toughness at cryogenic temperatures.

The second phase of the program was therefore aimed at making a quantitative determination of the effects of variations in content of impurities, particularly oxygen and iron contents, on the properties of the Ti-5Al-2.5Sn alloy from 78 to −423 F. As a result of this phase of the investigation, a new specification, GD/A ES 0-71010, was prepared, which limited the iron and interstitial contents and thereby made a considerable improvement in the toughness of the alloy at −423 F.

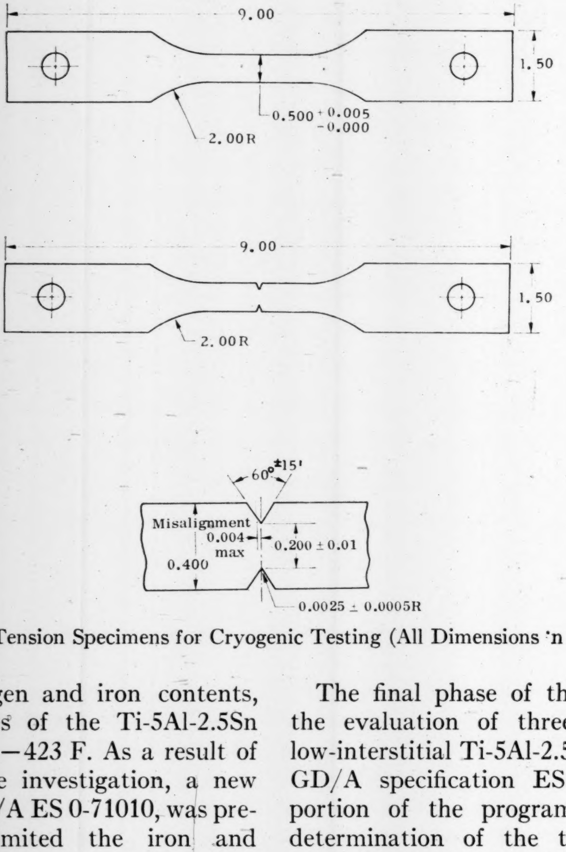


FIG. 1.—Tension Specimens for Cryogenic Testing (All Dimensions in Inches).

Decreasing the amount of iron and oxygen present in the alloy to the level necessary to ensure toughness at −423 F also resulted in a significant decrease in the yield and tensile strengths of the material at room temperature. To compensate for the decrease, some improvement in the ambient temperature strength of the low-interstitial alloy was sought by cold rolling. The objective of the third phase of the program was to determine quantitatively the effects of cold rolling on the strength and toughness of the material over the temperature range of 78 to −423 F.

The final phase of the program was the evaluation of three heats of the low-interstitial Ti-5Al-2.5Sn produced to GD/A specification ES 0-71010. This portion of the program involved the determination of the tensile, notched tensile, and fusion weld tensile properties as a function of the following factors: (1) testing temperature from 78 to −423 F, (2) thickness of sheet from 0.010 to 0.125 in., and (3) various conditions as to the temper of the metal such as mill annealed (as received), vacuum annealed, and stretch formed.

The information provided herein not only substantiates some of the conclusions of past investigations, but also adds new data to the literature on the effects of impurities and processing on the properties of the Ti-5Al-2.5Sn alloy. In addition, it provides preliminary design strength information on the low-interstitial Ti-5Al-2.5Sn alloy. The latter is believed to be the highest strength-to-density metal at −423 F which possesses adequate toughness for liquid hydrogen pressure vessel applications.

MATERIALS AND PROCEDURE

The condition and chemical analyses of the materials tested in this investigation are given in Table 1. The chemical analyses were provided by the suppliers or determined by the General Dynamics/Astronautics Analytical Laboratory. Hardness measurements were made on the Superficial Rockwell 15-N scale at room temperature. The materials were tested in the thicknesses and tempers shown in Table 1. The cold rolling was performed at room temperature on a 6-in. diameter 2-high laboratory rolling mill at General Dynamics/Astronautics, except for heat XT-60080, which was cold rolled by the supplier. The vacuum annealing (1450 F, for 1 hr) and stretch forming was also performed at General Dynamics/Astronautics. The stretch forming was performed over a production die having a compound radius of 30 by 60 in. which resulted in a maximum strain of about 8 per cent. All fusion butt welds were made by the tungsten-inert gas process with typical weld schedules, as given in reference (**8**). The welds were inspected radiographically to ensure that only sound welds were tested in the evaluation program.

The tension specimens used in this investigation are shown in Fig. 1. The stress concentration factor, K_t, for the notched tension specimens was 6.3 as determined by $K_t = \sqrt{a/r}$ where $a = \frac{1}{2}$ of the width between the notches and $r =$ the radii of the notches.

The testing apparatus consisted of universal testing machines equipped with continuous stress-strain recorders and strain pacers. Standard extensometers were used for room-temperature testing, and specially designed cryo-extensometers for low-temperature testing. The subzero temperature tests were performed in cryostats mounted on the universal testing machines. A full description of the cryostats, cryoextensometers, and accessory equipment, as well as safety features and rapidity of testing may be found in reference (**10**). The tension machines, extensometers, and accessory equipment were periodically checked and calibrated.

Tension tests were performed at 78 F (room temperature), at −100 F by immersion in a bath of dry ice and alcohol, at −320 F by immersion in liquid nitrogen, and at −423 F by immersion in liquid hydrogen. Tests were conducted after the specimens had reached equilibrium temperature, 2 to 4 min after immersion, as determined by copper-constantan thermocouples. The smooth tension specimens were tested at a strain rate of 0.001 in. per in. of gage length per min until yield, followed by 0.15 in. per min head speed until failure. Notched specimens were tested at 0.01 in. per min until failure. Yield strengths were determined from the stress-strain curves by the 0.2 per cent offset method. Elongations are reported as total elongations over a 2-in. gage length.

DISCUSSION OF RESULTS

The results obtained in this investigation are analyzed and discussed in four parts which represent the chronological order of testing.

TABLE 2.—RESULTS OF SCREENING TESTS ON Ti-5Al-2.5Sn ALLOY.

Heat	Test Temperature, deg F	Grain Direction[a]	F_{ty}, ksi	F_{tu}, ksi	Elongation in 2 in., per cent	Notch Tensile Strength (K_t = 6.3), ksi	Notched: Unnotched Tensile Strength Ratio	Weld Tensile Strength, ksi	Weld Elongation, per cent	Joint Efficiency, per cent
M-8465.....	78	L	113	118	19	158	1.34	121	12	100
		T	115	120	14	159	1.33	...	...	...
	−100	L	135	142	18	175	1.23	142	11	100
		T	137	144	11	...	...	...	...	...
	−320	L	184	196	15	226	1.15	192	8	98
		T	185	199	11	220	1.11	...	...	...
	−423	L	230	247	15	239	0.97	233	4	94
		T	230	244	11	208	0.85	...	...	...
M-9048.....	78	L	123	133	15	178	1.34	...	...	...
		T	124	132	14	177	1.34	...	...	...
	−100	L	146	160	15	195	1.22	...	...	...
		T	147	157	14	190	1.21	...	...	...
	−320	L	195	210	17	247	1.18	...	...	...
		T	198	212	14	238	1.12	...	...	...
	−423	L	238	259	9	209	0.81	...	...	...
		T	231	255	8	213	0.84	...	...	...
31387.......	78	L	111	123	12	151	1.23	121	9	98
	−423	L	239	253	4	217	0.86	267	2	100
D-2073......	78	L	122	130	17	174	1.34	...	...	...
		T	...	...	...	175	...	...	...	...
	−100	L	146	153	17	201	1.31	...	...	...
	−320	L	203	214	13	246	1.15	...	...	...
	−423	L	258	263	2	186	0.71	...	...	...
		T				164	...	...	...	...
D-2053......	78	L	126	141	14	172	1.22	...	...	...
		T	127	138	16	168	1.22	...	...	...
	−423	L	245	267	3	157	0.59	...	...	...
		T	229	248	2	157	0.63	...	...	...
M-8394.....	78	L	116	124	16	162	1.31	123	15	99
		T	117	123	13	162	1.32	120	14	98
	−100	L	135	145	13	176	1.21	146	14	100
		T	137	144	11	178	1.24	142	12	99
	−320	L	186	198	12	237	1.20	200	11	100
		T	188	199	8	235	1.18	196	11	98
	−423	L	234	250	5	218	0.87	245	5	98
		T	235	248	4	216	0.87	242	5	98
M-8613.....	78	L	128	130	22	...	...	...	...	...
	−423	L	240	262	4	161	0.62	...	...	...

[a] L = longitudinal, T = transverse.

Screening Tests:

Early in 1959, a program was initiated to evaluate the mechanical properties of a number of high-strength sheet materials from 78 to −423 F. Included in that program was the Ti-5Al-2.5Sn alloy. Since 1959 a total of seven heats of this alloy, produced commercially to AMS-4910 specifications, have been evaluated. The results are presented in Table 2. Figures 2 and 3 are graphical presentations of the data for the longitudinal test specimens.

As may be seen in Figs. 2 and 3, there appears to be a typical heat-to-heat scatter in the yield and tensile strengths

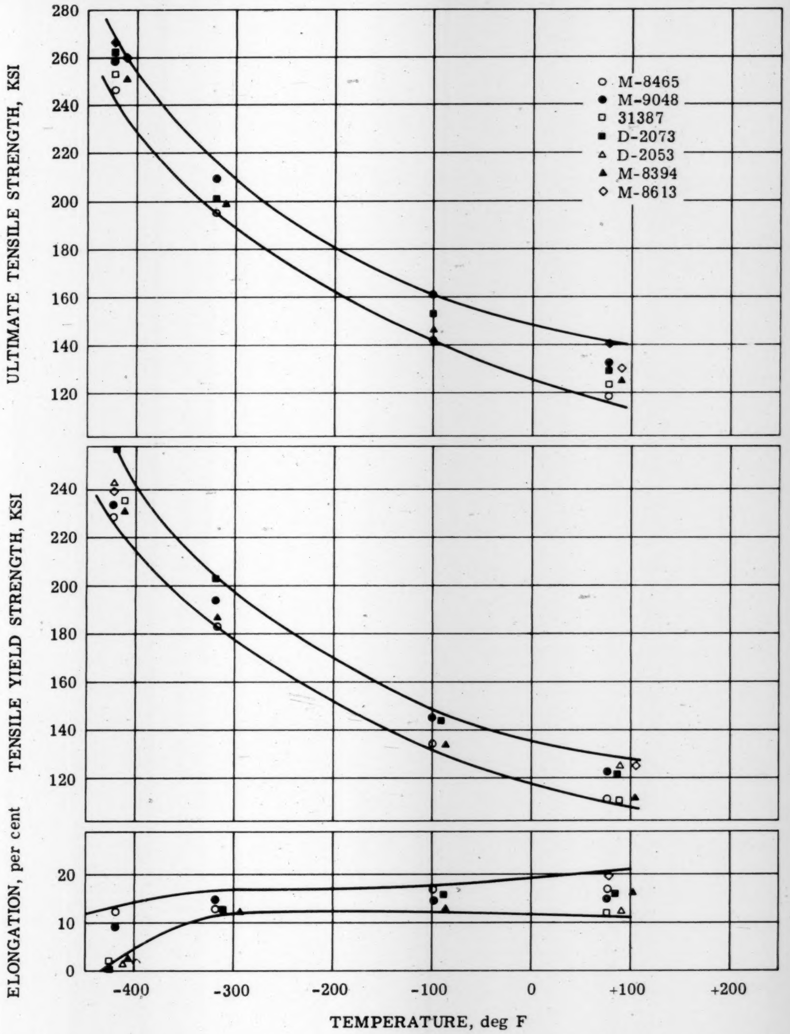


FIG. 2.—Tensile Properties Versus Temperature for Screening Materials (AMS 4910).

at each testing temperature. There is also an increase of approximately 100 per cent in both the yield and tensile strengths from 78 to −423 F.

The test data for tensile elongation, notched tensile strength, and resulting notched:unnotched tensile strength ratio exhibit a normal amount of scatter at 78, −100 and −320 F, as shown in Figs. 2 and 3; however, the results indicate an appreciable difference in these properties from heat to heat at −423 F. The first heat of material evaluated was M-8465, which exhibited quite good notched toughness at −423 F; however, because other investigations reported poor notched toughness of this alloy (**1**,**2**,**5**), several other heats were evaluated. Since a variety of notched tension specimens, with stress concentration factors ranging from 3.5 to 21 (**6**), have been used by the various investigators,

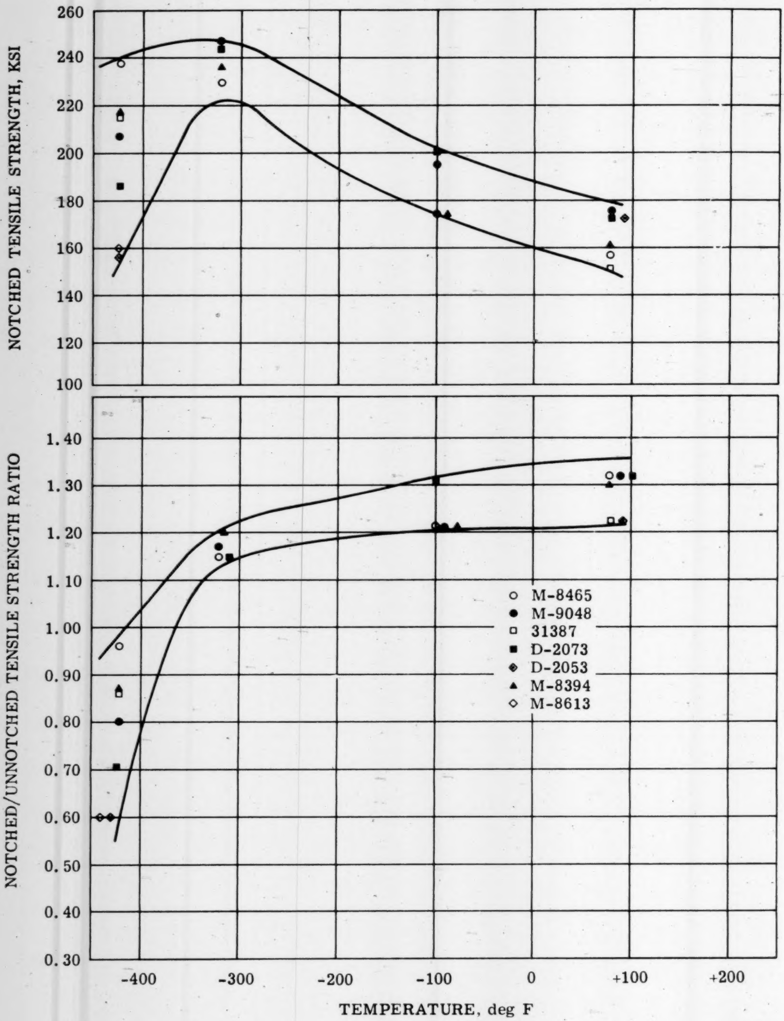


FIG. 3.—Notched Tensile Properties Versus Temperature for Screening Materials (AMS 4910).

it was considered desirable to evaluate the same heat of material by two different configurations of notched specimens in order to determine whether the discrepancies were due to valid differences in properties between the heats or due to the difference in notched specimens being used. Therefore heat D-2073, which also was evaluated by Espey, Jones, and Brown, using very sharp edge notched specimens (**5**), was tested in the present investigation. Although the absolute values for the notched tensile strength and notched: unnotched tensile strength ratio were different for the two notched configurations, it was evident from the data that heat D-2073 was notch-sensitive and exhibited poor toughness at −423 F as compared to heat M-8465. It was there-

TABLE 3.—EFFECTS OF IMPURITIES (IRON AND INTERSTITIAL CONTENTS) ON MECHANICAL PROPERTIES OF Ti-5Al-2.5Sn ALLOY.

Heat	Impurities, weight per cent		Test Temperature, deg F	Grain Direction[a]	F_{ty}, ksi	F_{tu}, ksi	Elongation in 2 in., per cent	Notch Tensile Strength ($K_t = 6.3$), ksi	Notched: Unnotched Tensile Strength Ratio
	Iron	Oxygen							
D-1311[b] (0.036 in.)	0.27	0.20	−423	L	256	265	7	166	0.65
V-1866[b] (0.035 in.)	0.50	0.08	−423	L	226	252	6	180	0.71
V-1867[b] (0.041 in.)	0.15	0.07	−423	L	211	232	14	233	1.00
D-1311 (0.020 in.)	0.27	0.20	78	L	115	128	17	167	1.30
				T	116	127	16	166	1.31
			−423	L	234	252	7	212	0.84
				T	228	241	1	195	0.81
V-1866 (0.020 in.)	0.50	0.08	78	L	109	121	16	147	1.21
				T	111	122	17	151	1.24
			−423	L	217	234	9	213	0.91
				T	219	238	5	182	0.77
V-1867 (0.020 in.)	0.15	0.07	78	L	99.8	111	18	152	1.37
				T	99.5	110	18	151	1.37
			−423	L	211	230	17	258	1.12
				T	204	227	11	238	1.05
V-1699	...	0.09	78	L	106	115	16	154	1.34
			−320	L	169	181	17	227	1.25
			−423	L	198	223	13	254	1.14
V-1668	...	0.11	78	L	102	112	17	155	1.38
			−320	L	168	180	16	228	1.27
			−423	L	206	229	15	253	1.10
V-1669	...	0.15	78	L	105	116	17	157	1.35
			−320	L	173	186	15	231	1.24
			−423	L	214	230	11	234	1.02
V-1670	...	0.17	78	L	107	118	16	161	1.36
			−320	L	177	191	15	234	1.23
			−423	L	225	237	13	216	0.91
V-1671	...	0.24	78	L	117	123	17	172	1.40
			−320	L	188	201	15	236	1.17
			−423	L	238	252	5	196	0.78

[a] L = longitudinal; T = transverse.

[b] Hand rolled; remaining data on same heats were obtained on strip-rolled material.

fore felt that the large scatter in elongation and notched properties at −423 F (see Figs. 2 and 3) was due to variations in the material from heat to heat.

Since all of the screening heats were evaluated in the hot-rolled plus mill annealed condition, it was concluded that chemical composition, and not condition or processing history, was responsible for the heat-to-heat variations in toughness at −423 F. Based upon past studies (**3**,**4**), it was suspected that the interstitial elements (carbon, oxygen, nitrogen, hydrogen) were responsible for low-temperature embrittlement. As may be seen in Tables 1 and 2, those heats with the lowest amounts of interstitial elements possessed the best

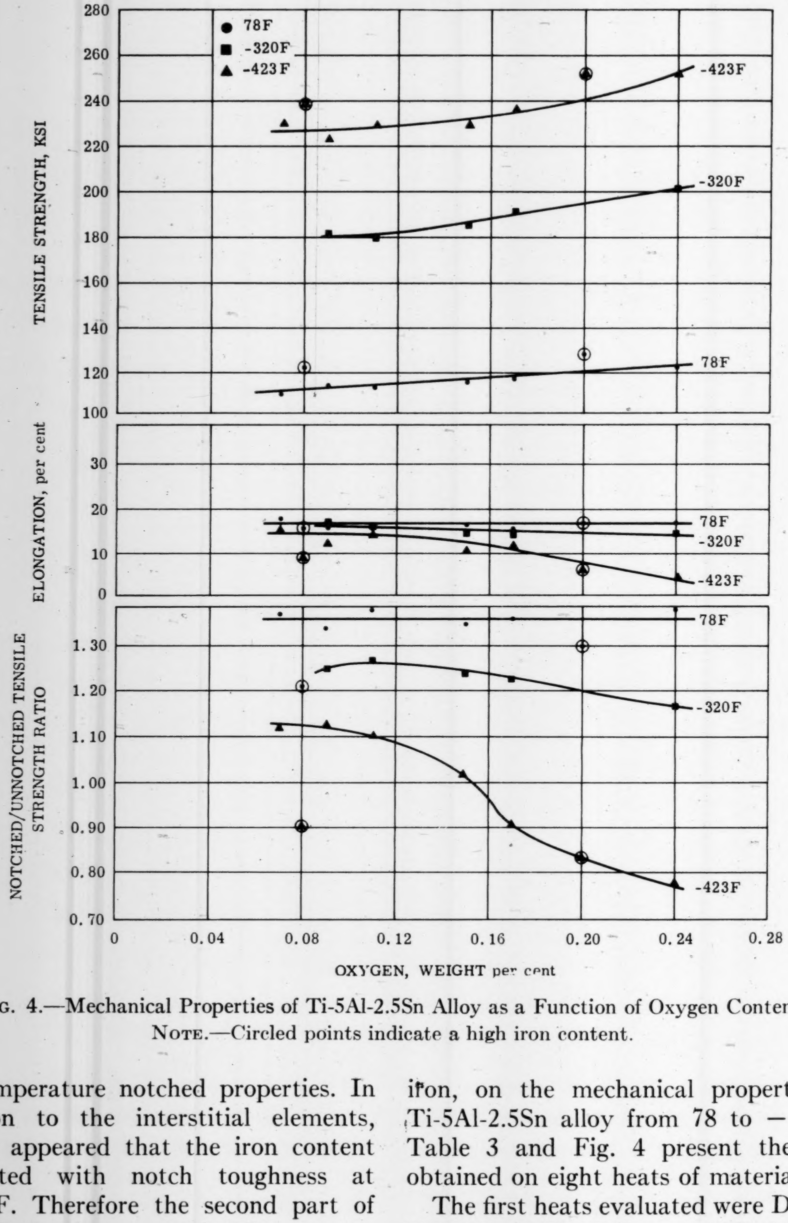


FIG. 4.—Mechanical Properties of Ti-5Al-2.5Sn Alloy as a Function of Oxygen Content.
NOTE.—Circled points indicate a high iron content.

low-temperature notched properties. In addition to the interstitial elements, it also appeared that the iron content correlated with notch toughness at −423 F. Therefore the second part of the investigation was undertaken.

Effects of Impurities:

The purpose of the second phase of the program was to evaluate the effect of impurities, in particular oxygen and iron, on the mechanical properties of Ti-5Al-2.5Sn alloy from 78 to −423 F. Table 3 and Fig. 4 present the data obtained on eight heats of material.

The first heats evaluated were D-1311, V-1866 and V-1867, as supplied in the hand-rolled plus mill annealed condition. These same heats were later strip-rolled and evaluated. It was evident from the data that the low-oxygen - low-iron heat possessed superior notched proper-

TABLE 4.—EFFECTS OF COLD ROLLING ON MECHANICAL PROPERTIES OF TITANIUM-5Al-2.5Sn ALLOY.

Heat	Cold Rolling, per cent reduction	Test Temperature, deg F	Grain Direction[a]	F_{ty}, ksi	F_{tu}, ksi	Elongation in 2 in., per cent	Notched Tensile Strength ($K_t = 6.3$), ksi	Notched: Unnotched Tensile Strength Ratio
M-9048.......	0	78	L	123	133	15	178	1.34
			T	124	132	14	177	1.34
		−423	L	238	259	9	209	0.81
			T	231	255	8	213	0.84
	12	78	L	138	161	8	208	1.29
		−423	L	256	284	6	208	0.73
M-8465.......	0	78	L	113	118	19	158	1.34
			T	115	120	14	159	1.33
		−423	L	230	247	15	239	0.97
			T	230	244	11	208	0.85
	20	78	L	125	145	9	189	1.30
		−423	L	244	274	4	230	0.84
	30	78	L	129	154	1	136	0.88
		−423	L	...	...	...	140	...
	42	78	L	131	...	1	101	...
		−423	L	...	...	...	110	...
XT-60080.....	0	78	L	83.8	94.9	17	126	1.33
			T	86.3	94.9	17	127	1.34
		−423	L	190	208	12	234	1.12
			T	190	209	13	234	1.12
	10	78	L	99.7	116	10	153	1.32
			T	98.4	116	11	152	1.31
		−423	L	215	236	7	274	1.16
			T	208	234	8	267	1.14
	20	78	L	103	120	8	147	1.22
			T	97.8	120	9	157	1.30
		−423	L	198	237	6	237	1.00
			T	202	232	6	264	1.13
	30	78	L	106	123	7	139	1.13
			T	99.2	122	9	158	1.29
		−423	L	198	231	3	177	0.77
			T	206	233	9	257	1.10
	40	78	L	108	127	7	134	1.05
			T	...	129	8	162	1.25
		−423	L	207	238	4	164	0.69
			T	212	236	9	249	1.05
	50	78	L	107	127	5	118	0.93
			T	107	126	8	159	1.26
		−423	L	203	229	1	143	0.63
			T	212	234	9	223	0.95

[a] L = longitudinal; T = transverse.

ties at −423 F than did the medium-oxygen - medium-iron or low-oxygen - high-iron heats. This is attributed, at least in part, to the large amount of a second phase present in the latter two heats (see Fig. 8). Identity of these particles in the microstructure remains in doubt. They are believed to be either beta phase or an intermetallic composed of Ti-Fe or Ti-Al-Fe.

In addition to the three heats mentioned above, a series of five heats of Ti-5Al-2.5Sn alloy with oxygen contents varying from 0.09 to 0.24 was then evaluated. Based on the test data for these eight heats, it appears that an oxygen

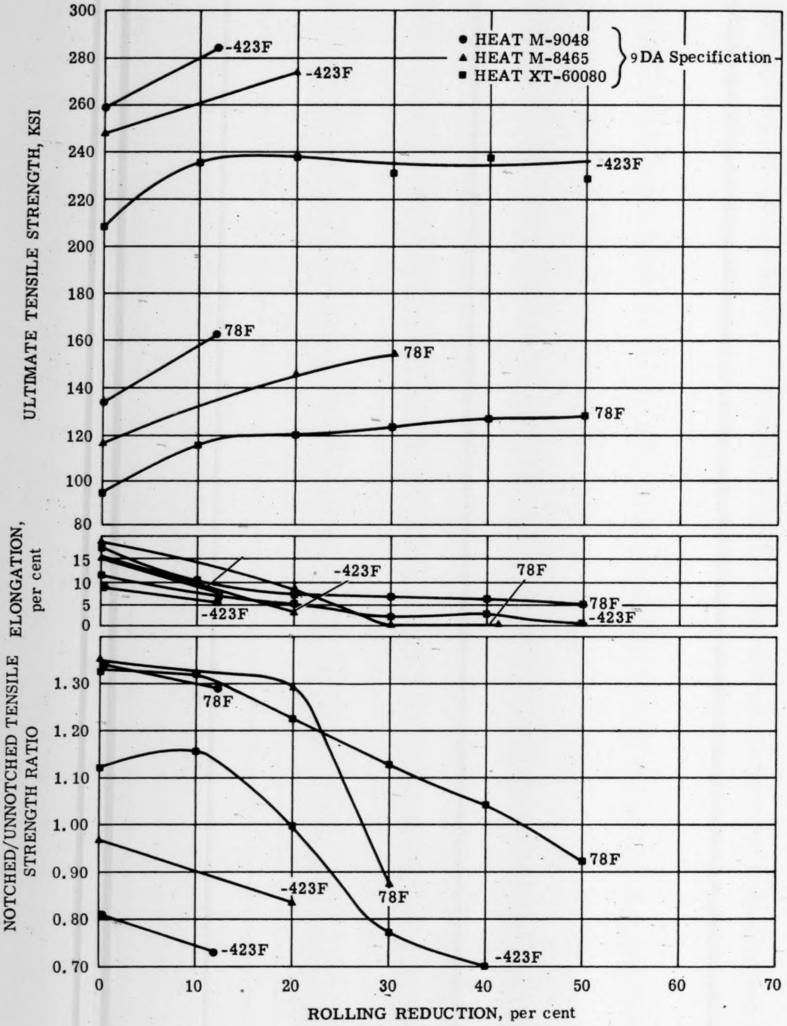


Fig. 5.—Mechanical Properties of Ti-5Al-2.5Sn Alloy as a Function of Per Cent Cold Work.

content above about 0.12 per cent causes embrittlement of the alloy at liquid hydrogen temperature.

Analysis of the data (see Fig. 4) shows that increasing the oxygen and iron contents results in an increase of approximately 10 per cent in the tensile strengths at each of the testing temperatures. This is attributed to an increase in the amount of beta or intermetallic phase present and to strengthening due to the interstitial solid solution, that is, the blocking of dislocations at areas where oxygen atoms are located in the lattice as interstitials.

Increasing the oxygen content beyond about 0.12 weight per cent does not severely affect the ductility (elongation) or toughness (notched properties) at 78 or −320 F, but does cause a signifi-

TABLE 5.—PROPERTIES OF Ti-5Al-2.5Sn ALLOY PRODUCED TO GD/A—0-71010 SPECIFICATION.

Heat	Test Temperature, deg F	Direction[a]	F_{ty}, ksi	F_{tu}, ksi	Elongation in 2 in., per cent	Notched Tensile Strength ($K_t = 6.3$), ksi	Notched: Unnotched Tensile Ratio	Weld Tensile Strength, ksi	Weld Elongation, per cent	Joint Efficiency, per cent
3930131 (mill annealed)	78	L	117	127	15	168	1.32	114	2	90
		T	118	126	14	168	1.33	117	2	93
	−320	L	175	189	18	233	1.23	183	2	97
		T	182	190	17	243	1.28	179	2	94
	−423	L	225	239	13	260	1.09	222	2	93
		T	226	235	11	247	1.05	214	1	91
3930131 (vacuum annealed)	78	L	89.0	105	16	141	1.34	...	...	...
	−320	L	170	182	13	221	1.21	...	...	...
	−423	L	206	219	3	222	1.01	...	...	...
3930131 (stretch formed)[b]	78	L	125	133	12	...	...	...	...	...
	−320	L	199	200	13	246	1.23	...	...	...
	−423	L	240	248	7	254	1.02	...	...	...
3930131 (stretch formed[b] followed by vacuum annealed)	78	L	90.6	108	17	136	1.25	...	...	...
	−320	L	171	183	11	223	1.22	...	...	...
	−423	L	223	236	3	232	0.98	...	...	...
3960328 (0.017 in., mill annealed)	78	L	105	114	17	142	1.24	...	...	...
		T	103	113	15	153	1.36	...	...	...
	−100	L	122	134	16	161	1.20	...	...	...
		T	122	133	16	176	1.32	...	...	...
	−320	L	169	182	17	210	1.15	...	...	...
		T	165	177	17	228	1.29	...	...	...
	−423	L	209	226	14	207	0.92	...	...	...
		T	208	226	15	214	0.95	...	...	...
3960328 (0.017 in., stretch formed[b])	78	L	107	114	14	149	1.31	...	...	...
		T	101	113	15	150	1.33	...	...	...
	−100	L	130	134	13	171	1.27	...	...	...
		T	121	133	13	174	1.31	...	...	...
	−320	L	175	185	17	223	1.20	...	...	...
		T	168	185	16	223	1.20	...	...	...
	−423	L	215	230	12	223	0.97	...	...	...
		T	203	231	9	221	0.96	...	...	...
3960328 (0.025 in., mill annealed)	78	L	98.0	109	17	138	1.26	104	14	95
		T	100	110	17	150	1.36	...	...	...
	−100	L	117	129	16	159	1.23	131	13	100
		T	120	130	16	171	1.31	...	...	...
	−320	L	162	184	17	209	1.14	183	12	100
		T	170	180	17	223	1.24	...	...	...
	−423	L	201	220	13	205	0.93	210	2	95
		T	203	226	16	219	0.97	...	...	...
3960328 (0.063 in., mill annealed)	78	L	104	122	14	152	1.25	114	7	93
		T	107	114	13	158	1.39	...	...	...
	−100	L	125	144	16	180	1.25	141	8	98
		T	131	140	11	187	1.34	...	...	...
	−320	L	173	194	17	229	1.18	190	5	98
		T	179	189	12	235	1.24	...	...	...
	−423	L	209	239	14	228	0.95	225	...	94
		T	214	242	9	243	1.00	...	...	...

TABLE 5.—*Concluded.*

Heat	Test Temperature, deg F	Direction[a]	F_{ty}, ksi	F_{tu}, ksi	Elongation in 2 in., per cent	Notched Tensile Strength ($K_t = 6.3$), ksi	Notched: Unnotched Tensile Ratio	Weld Tensile Strength, ksi	Weld Elongation, per cent	Joint Efficiency, per cent
3960328 (0.125 in., mill annealed)...	78	L	121	137	12	167	1.22	...	...	...
		T	126	137	13	164	1.20	...	...	...
	−100	L	143	163	13	186	1.14	...	...	...
		T	149	163	12	190	1.16	...	...	...
	−320	L	187	208	15	233	1.12	...	...	...
		T	192	204	21	212	1.04	...	...	...
	−423	L	241	248	5	199	0.80	...	...	...
		T	221	229	10	177	0.77	...	...	...
3930498 (0.015 in., mill annealed)...	78	L	113	121	12	160	1.32	...	...	...
		T	110	117	14	161	1.37	...	...	...
	−100	L	133	143	11	183	1.28	...	...	...
		T	131	140	12	184	1.31	...	...	...
	−320	L	181	189	...	238	1.26	...	...	...
		T	178	186	17	239	1.28	...	...	...
	−423	L	226	231	...	238	1.03	...	...	...
		T	217	231	12	248	1.07	...	...	...
3930498 (0.010 in., mill annealed)...	−320	L	185	194	...	...	...	...	...	...
		T	178	186	14	...	...	...	...	...

[a] L = longitudinal; T = transverse.

[b] Stretch formed material reduced 3 to 6 per cent in thickness.

cant decrease in these properties at −423 F. It is also evident from the data that large amounts of iron in the Ti-5Al-2.5Sn alloy results in a decrease in the toughness at −423 F.

From these data, a specification was prepared (GD/A Specification 0-71010) which limits the oxygen content to 0.12 and the iron content to 0.25 weight per cent maxima. As a result of decreasing the normal oxygen and iron contents, the yield and tensile strengths were also decreased. In an effort to restore the strength of the low-interstitial Ti-5Al-2.5Sn, a cold-rolling program was initiated.

Effects of Cold Rolling:

The purpose of this phase of the program was to determine the effect of cold rolling on the mechanical properties of the Ti-5Al-2.5Sn alloy at room and liquid hydrogen temperatures. The results are given in Table 4 and Fig. 5.

Three heats of material were tested. Two heats, M-8465 and XT-60080, conformed to the GD/A Specification 0-71010. The results show that a substantial increase in yield and tensile strengths at 78 and −423 F could be obtained by cold rolling 10 to 20 per cent; however, cold rolling beyond 20 per cent reduction did not result in an appreciable further increase of strength. It is also apparent (Fig. 5) that cold rolling decreased the elongation and notched toughness. It appears that rolling reductions in excess of 10 per cent cause severe reduction in toughness at −423 F. It should also be mentioned that an unavoidable result of the use of cold rolling to obtain strength in the sheet is the necessity to reinforce welded joints; that is, the weld will possess only the same strength as the annealed metal. Specification 0-71010 permits final skin-pass rolling, not to exceed 0.001 in.

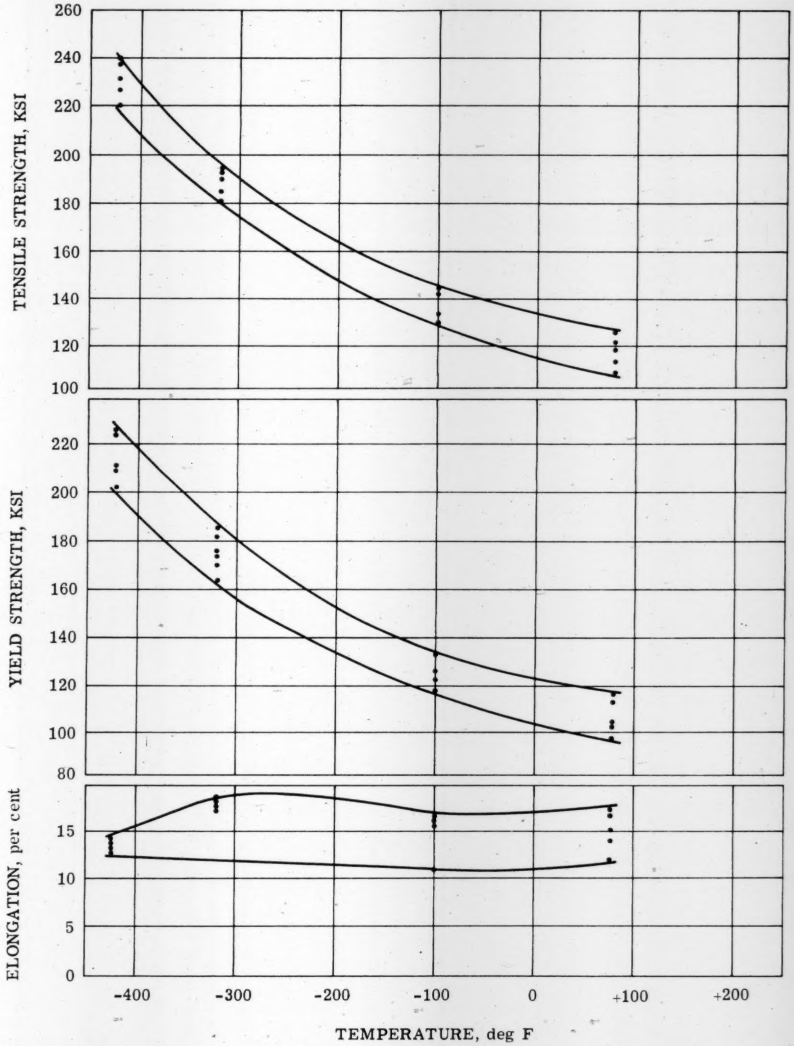


FIG. 6.—Tensile Properties Versus Temperature for Three Heats of Ti-5Al-2.5Sn Alloy, GD/A Specification 0-71010.

reduction, for the purpose of improving the surface and flatness of the sheet.

Properties of Low-Impurity Ti-5Al-2.5 Sn:

The purpose of this part of the program was to evaluate the Ti-5Al-2.5Sn alloy produced to the GD/A Specification 0-71010 to determine its feasibility for use in very thin-walled pressure-stabilized vehicle tanks at liquid hydrogen temperatures. Three heats of material which varied in thickness from 0.010 to 0.125 in. were evaluated. The alloy was tested in the as-received, vacuum annealed, and stretch formed conditions. The results are presented in

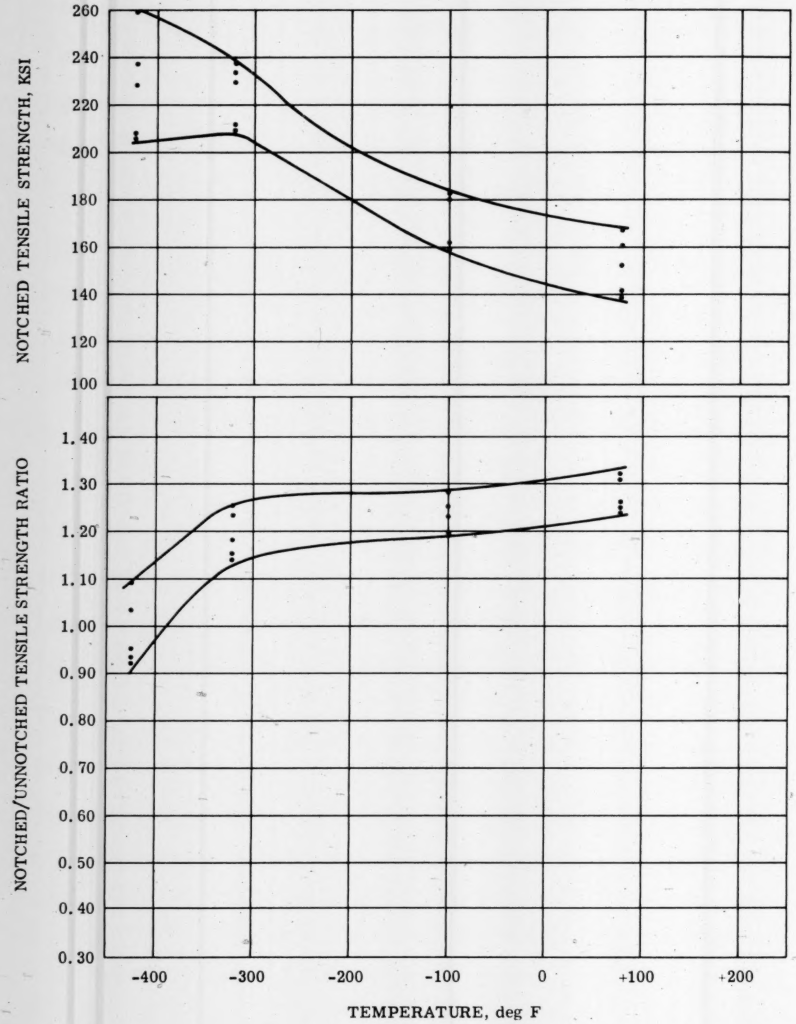
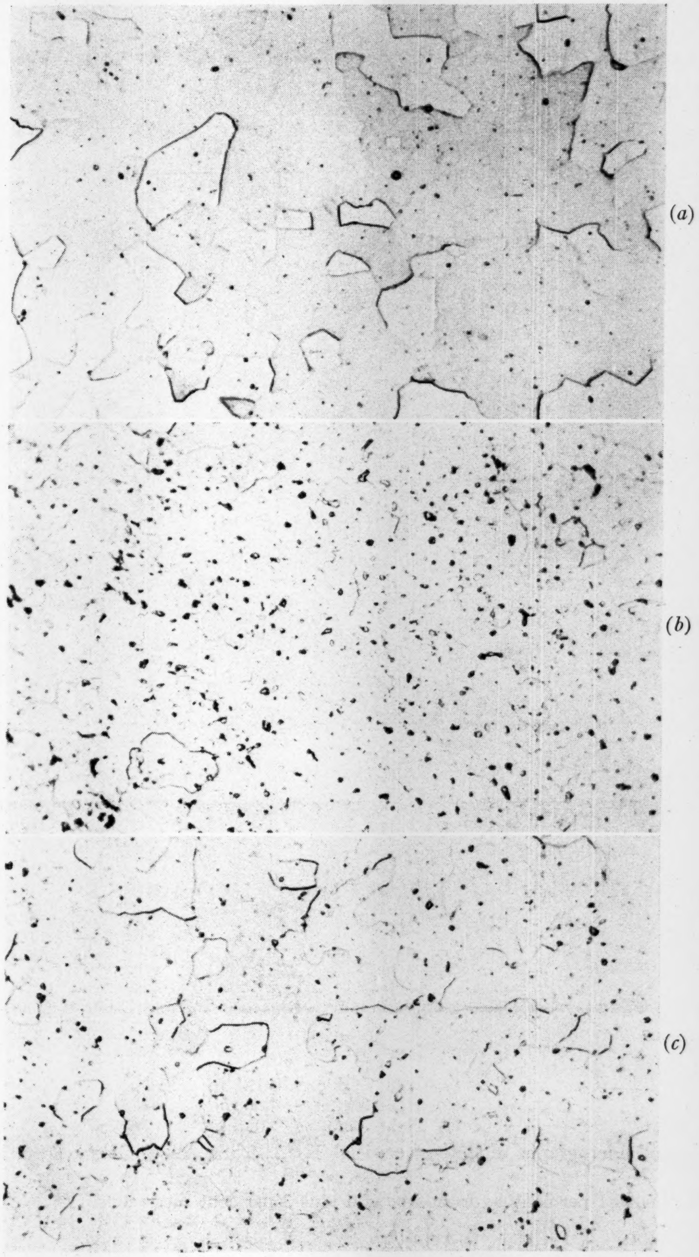


FIG. 7.—Notched Tensile Properties Versus Temperature for Three Heats of Ti-5Al-2.5Sn Alloy, GD/A Specification 0-71010.

Table 5 and Figs. 6 and 7; photomicrographs are shown in Fig. 9.

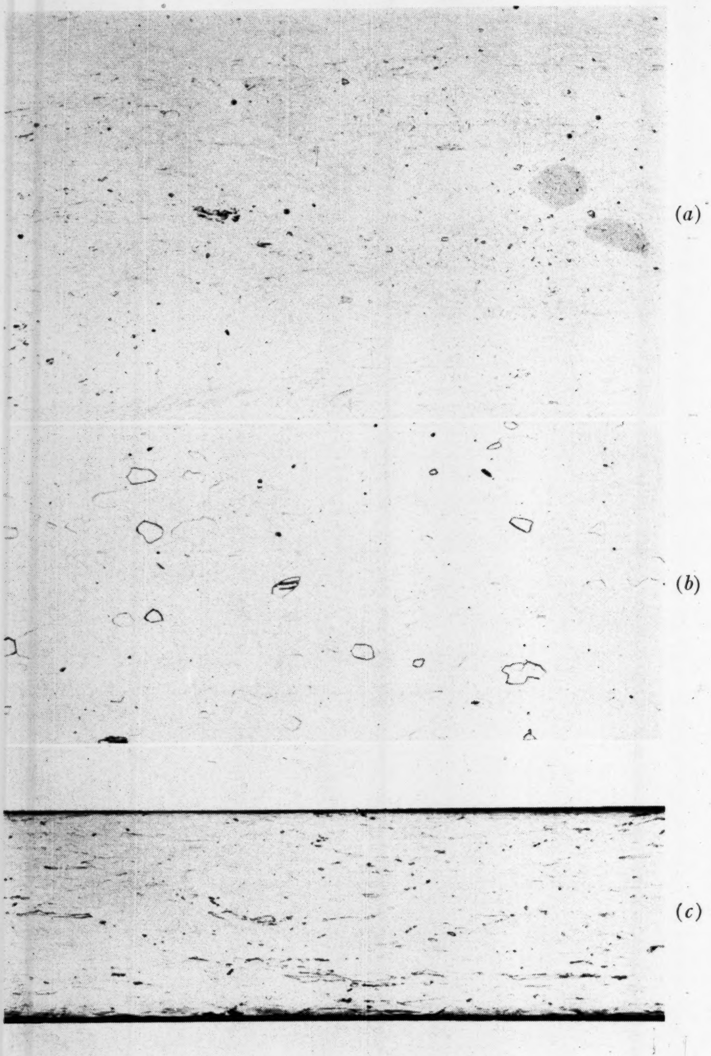
As compared to the data obtained from the screening tests, the low-impurity Ti-5Al-2.5Sn alloy exhibited somewhat lower yield and tensile strengths but also possessed significantly better notched properties at −423 F. Although there is still evidence of a decrease in the notched toughness from −320 to −423 F, the value of the notched:unnotched tensile strength ratio is near unity or above (0.92 to 1.09) with one exception, the 0.125 in. thick Ti-5Al-2.5Sn alloy at −423 F. This is a significant improvement as compared to the values of 0.59 to 0.97 for the notched: unnotched tensile strength ratio of the



(*a*) Heat V-1867; oxygen, 0.07 per cent, iron, 0.15 per cent.
(*b*) Heat V-1866; oxygen, 0.08 per cent, iron, 0.50 per cent.
(*c*) Heat D-1311; oxygen, 0.20 per cent, iron, 0.27 per cent.

FIG. 8.—Microstructures of Three Experimental Heats of Ti-5Al-2.5Sn Alloy Which Show That Amount of Second Phase Particles in the Alpha Matrix is Directly Related to Iron Content (×500).

Etchant, aqueous 1 per cent hydrofluoric acid plus 3 per cent nitric acid.



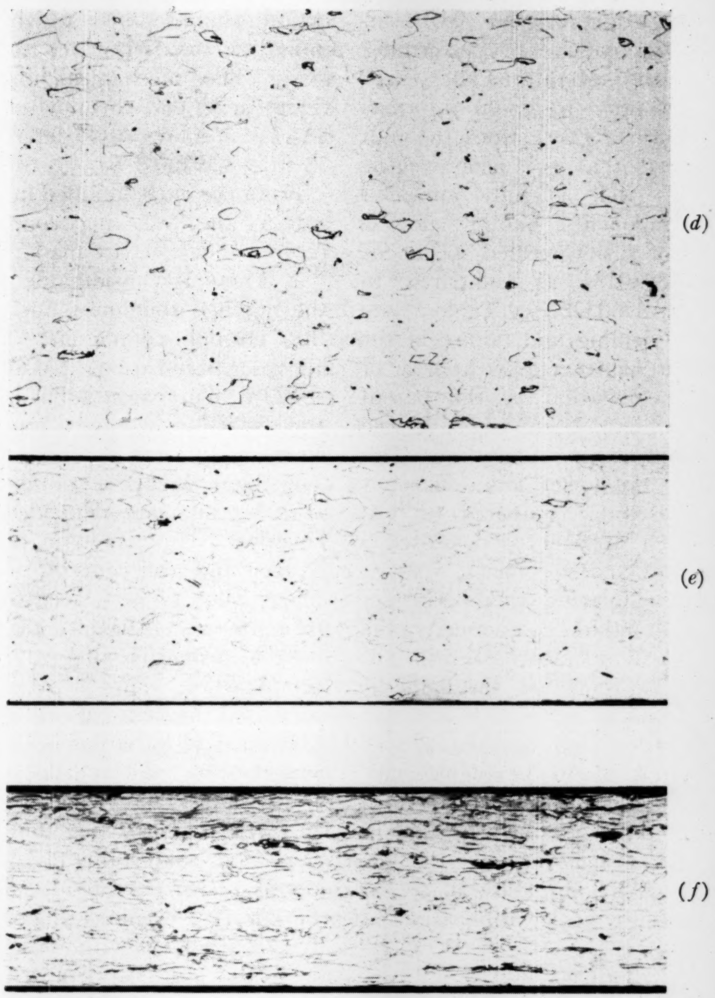
(a) Heat 3960328, 0.125 in.
(b) Heat 3960328, 0.063 in.
(c) Heat 3960328, 0.025 in.

FIG. 9.—Photomicrographs of Low-Interstitial Ti-5Al-2.5Sn Alloy, GD/A Specification 0-71010, As-Received (×75).

Etchant, aqueous 1 per cent hydrofluoric acid plus 3 per cent nitric acid.

normal commercial material at −423 F. There is also a considerable improvement in the elongation at −423 F for the heats and conditions tested.

All of the sheet evaluated in this part of the program was processed in the mill by cold rolling and intermediate annealing with the exception of the 0.125 in. thick sheet from heat 3960328. The latter material was hot rolled. The cause of the lower notched:unnotched tensile strength ratio of the 0.125 in.



(*d*) Heat 3960328, 0.017 in.
(*e*) Heat 3930131, 0.014 in.
(*f*) Heat 3930498, 0.015 in.

FIG. 9.—*Continued.*

thick material is believed to be due to the presence of the hot-rolled structure (see Fig. 9).

The lower strength of the vacuum annealed material and the less than 100 per cent joint efficiencies of the butt fusion welds in the as-received sheet indicate that the latter materials were not fully annealed by the final strip-annealing method used at the mill. As indicated above, final rolling to improve flatness is permitted up to

0.001 in. reduction (that is, as much as 10 per cent reduction) for 0.010 in. material. One objective of performing the tests on the several thicknesses of sheet was to provide design information for structures made from the mill annealed sheet, that is, fusion welded structures, as well as fully annealed parts and components. The low elongation values for fusion welded specimens from heat 3930131 as compared to those for heat 3960328 (see Table 5) are due to local yielding and failure in the annealed weld zone area with little or no uniform elongation in the parent metal which had been slightly cold rolled as a flattening operation. This also explains the lower joint efficiency of heat 3930131 as compared to heat 3960328 which apparently possessed a lesser degree of cold working.

The stretch forming was performed over a 30 by 60-in. compound radius form at room temperature. Tensile and yield strengths remained the same or increased slightly.

The elongation and notched properties do not seem to be significantly affected by stretch forming.

From the data obtained in this investigation, it is believed that the low-impurity Ti-5Al-2.5Sn alloy is acceptable for pressure vessel applications at −423 F.

SUMMARY AND CONCLUSIONS

The tensile, notched tensile, and fusion weld tensile properties were determined from 78 to −423 F on a number of heats of the Ti-5Al-2.5Sn alloy. In addition to the determination of the properties of normal commercial heats, the investigation included the determination of the effects of impurities, primarily oxygen and iron contents, and the effects of cold rolling on the mechanical properties of Ti-5Al-2.5Sn at room and liquid hydrogen temperatures. From these data, a special specification was prepared which limits the interstitial and iron contents in the alloy. The mechanical properties of three heats of the low-impurity Ti-5Al-2.5Sn alloy were determined from 78 to −423 F.

From the data obtained in this investigation, the following conclusions and recommendations are made:

1. There is considerable variation in the notched toughness and ductility of the normal commercial Ti-5Al-2.5Sn alloy (Specification AMS 4910) at −423 F. This variation is attributed to the variable content of interstitial elements and iron.

2. The strength, notch toughness, and ductility are dependent upon the chemical composition, in particular the oxygen and iron content, of the Ti-5Al-2.5Sn alloy. The strength is affected from 78 to −423 F, whereas the toughness is primarily affected at cryogenic temperatures. This is attributed to an increased amount of a second phase, presumed to be either beta phase or an intermetallic, and to dislocation blocking by the interstitial elements with increase in the iron and oxygen contents.

3. Cold rolling the Ti-5Al-2.5Sn alloy up to 10 per cent reduction appears to be effective in increasing the yield and tensile strengths without severely affecting the low-temperature toughness. Cold rolling beyond about 20 per cent does not appear to cause significant further increases in strength, but reductions somewhat greater than 10 per cent do cause low-temperature embrittlement.

4. The low-impurity Ti-5Al-2.5Sn alloy (GD/A Specification 0-71010) appears to be acceptable for structural application to liquid hydrogen temperatures. The low-interstitial material exhibits excellent toughness and strength-to-density ratio from 78 to

−423 F in the as-received, stretch formed, or fusion welded conditions.

5. It is suggested that further studies similar to the present one, but conducted on higher strength titanium alloys such as Ti-6Al-4V and Ti-8Al-2Cb-1Va, may also be fruitful.

Acknowledgments:

The authors wish to acknowledge the assistance of their associates who contributed to this paper. Also, the authors wish to thank Crucible Steel Corp., Reactive Metals, Inc., Republic Steel Corp., and Titanium Metals Corp. of America for supplying some of the test materials. The work was performed under the sponsorship of General Dynamics/Astronautics, whose permission to publish this paper is gratefully acknowledged.

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ELEVATED-TEMPERATURE BEHAVIOR OF ANNEALED 70-30 COPPER-NICKEL

By Matthew J. Donachie, Jr.,[1] Roger K. Steele,[2] and Robert G. Shepheard[3]

Synopsis

Elevated-temperature tensile, hardness, and creep-rupture data have been developed on an annealed 70-30 copper-nickel alloy. The results suggest good agreement with the limited data available on this alloy composition in the annealed condition. Differences in strength level between alloys corresponding to the same specification suggest that composition limits of ASTM Specifications B 122 should be tightened.

Copper-nickel alloy tubes of 70-30 composition are widely used for such mildly elevated-temperature applications as condenser tubes and other unfired pressure vessels. Generally, tubes in the annealed temper are used, and in service the alloy normally does not see temperatures much above 300 F for any extended duration. Design factors for such annealed tubes have been largely based on the data of Burghoff et al (**1**,**2**)[4] (see also reference (**3**)) which is limited to temperatures below 500 F. The creep-rupture properties of 70-30 copper-nickel alloys have also been studied by Jenkins et al (**4**,**5**) and Simmons et al (**6**). References (**5**) and (**6**) pertain to cold-drawn material at temperatures up to 1200 F. Tests of cold-drawn 70-30 copper-nickel suggest that it may be advantageous for unfired pressure vessel applications up to 850 to 900 F (**6**). Results for annealed 70-30 copper-nickel have been less conclusive in establishing an upper limit for such applications, principally because of limited data available at temperatures of 500 F and above. (Table UNF 23, ASME Boiler and Pressure Vessel Code, Section VIII does, however, list allowable stresses for 70-30 copper-nickel to 700 F.) The data of Burghoff et al (**1**,**2**) leave much undetermined concerning the creep-rupture behavior of 70-30 copper-nickel even in the temperature range 300 to 500 F. For example, their data suggest that the slope of the log stress versus log rupture life plot is essentially flat at 400 F and leaves uncertain the slope of such a plot at higher temperatures.

The scarcity of data and gaps in existing data described above precipitated the elevated-temperature testing of annealed 70-30 copper-nickel described in this paper. The purpose of these tests was to generate mechanical property data which would aid in the evaluation of the useful range of application of this alloy.

Experimental Details

Material:

All specimens were machined from 1-in. diameter round bar stock in the

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[1] Pratt & Whitney Aircraft, East Hartford, Conn.

[2] Anaconda American Brass Co., Waterbury, Conn.

[3] International Business Machines Co., Poughkeepsie, N. Y.

[4] The boldface numbers in parentheses refer to the list of references appended to this paper.

TABLE 1.—COMPOSITION AND PROPERTIES OF TESTED ALLOY.

Composition:	
Copper	68.02
Nickel	30.84
Iron	0.57
Manganese	0.40
Zinc	<0.01
Lead	0.001
Room-Temperature Tensile Properties:	
Ultimate tensile strength, psi	64 100
Yield strength (0.5 per cent plastic strain), psi	32 000
Elongation in 2 in., per cent	34.5
Reduction of area, per cent	49.1
Grain Size:	
Grain size, mm	0.007

TABLE 2.—CREEP-RUPTURE TEST RESULTS.

Temperature, deg F	Stress, psi	Specimen	Time for Rupture, hr	Elongation in 1.8 in., per cent	Reduction of Area, per cent	Minimum Creep Rate, in. per in. per hr
500	55 000	CN-13	[a]	33.5	72	...
	54 000	CN-22	[a]	33.5	75	...
	53 000	CN-25	[a]	34.5	75	...
	52 500	CN-15	>3 000[b]	12.1[b]	16[b]	1.61×10^{-6}
	50 000	CN-2	14 322	running		6.83×10^{-7}
650	53 000	CN-8	[a]	33.5	76	...
	50 000	CN-11	[a]	31.0	73	...
	47 500	CN-14	121.0	26.0	28	8.40×10^{-4}
	45 000	CN-12	106.1	20.0	29	4.23×10^{-4}
	40 000	CN-3	4 016	14.0	19	1.45×10^{-5}
800	35 000	CN-5	7.6	11.0	20	6.01×10^{-3}
	32 500	CN-17	12.8	12.0	12	...
	30 000	CN-4	48.5	14.0	18	1.04×10^{-3}
	25 000	CN-6	167.2	1.0	17	1.88×10^{-4}
	22 500	CN-9	3 767	8.0	9	1.54×10^{-5}
950	22 500	CN-21	3.3	9.8	15	...
	21 000	CN-23	10.1	11.0	13	...
	20 000	CN-20	35.8	10.5	12	1.88×10^{-2}
	18 000	CN-16	38.3	7.0	13	1.47×10^{-3}
	15 000	CN-19	1 048.0	17.5	20	1.03×10^{-4}

[a] Broke on loading.

[b] Test stopped after 3000 hr due to temperature controller failure. Ductility values are from unbroken test piece.

mill-annealed condition. The stock conformed to Military Specification MIL-C-15726. This specification falls within the composition limits of ASTM Specifications B 122.[5] The chemical composition and tensile properties of the tested material are given in Table 1.

Procedures:

Creep-rupture, hot-hardness, and tension tests were performed. The creep-rupture and tension specimens were standard round 0.357-in. diameter reduced-section (0.1 sq in. area) tension bars. Creep-rupture tests were run on first-class lever machines having a load ratio of 20:1. Relative creep data were obtained from dial gages which recorded the vertical motion of the upper pull rod. Hardness testing was carried out in argon on a dead weight loaded hot-hardness tester constructed at General Dynamics/Electric Boat. Details of this machine appear elsewhere (**7**,**8**). Hardness specimens ($\frac{1}{4}$-in. thick flat disks 1 in. in diameter) were cut from the same stock used in creep rupture and tension testing. Hardness impressions were made with a 30-sec load application to a sapphire 136-deg pyramid indenter. Tension tests were conducted on a screw-driven tension machine in general accordance with ASTM Recommended Practice E 21.[6] However, a constant crosshead velocity of 0.2 in. per min (on ≈ 2-in. gage length) was used throughout the test and plastic strains under load were calculated from time-base recordings of the crosshead motion.

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[5] Specifications for Copper-Nickel-Zinc Alloy (Nickel Silver) and Copper-Nickel Alloy Plate, Sheet, Strip, and Rolled Bar (B 122), 1961 Book of ASTM Standards, Part 2.

TABLE 3.—VICKERS HARDNESS AT TEMPERATURE.

Temperature, deg F	Vickers Hardness
400	79.0
600	70.0
700	70.0
800	66.9
900	62.8
1000	59.3
1100	45.6
1200	36.3

TEST RESULTS

A total of 20 creep-rupture tests were carried out at four temperatures from 500 to 950 F. Hot-hardness tests were carried out in the temperature range 400 to 1200 F, and tension tests were carried out from room temperature to 1100 F. The creep-rupture data obtained are summarized in Table 2. Log-log plots of stress versus rupture life and versus minimum creep rate are shown in Fig. 1. Hardness data are given in Table 3 and plotted as Vickers hardness number (VHN) versus temperature in Fig. 2. Tensile data are given in Table 4 and engineering stress-strain curves at various temperatures are plotted in Fig. 3.

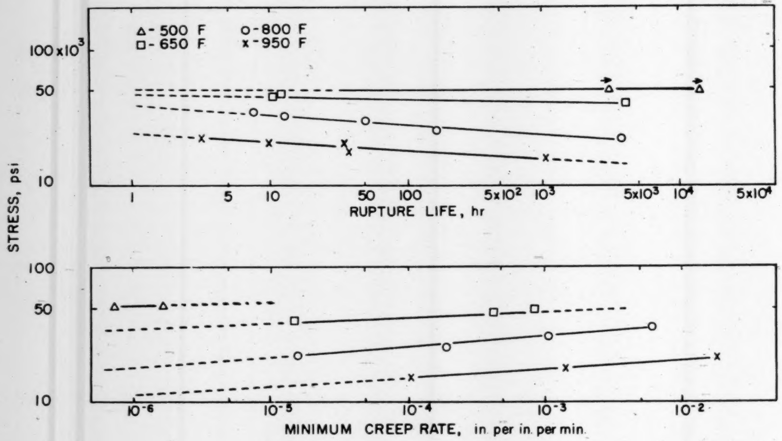

FIG. 1.—Creep-Rupture Results.

DISCUSSION

The log-log plot of stress versus rupture life indicates essentially linear behavior over the rupture life range from about 4 to 4000 hr. At 500 and 650 F, a flat or shallow slope is observed, in agreement with the work of Burghoff et al (1–3) which indicated that the stresses for a rupture life of 10^2, 10^3, 10^4, or 10^5 hr were identical at 400 F. The plotted data at 800 and 950 F showed definite slopes more characteristic of elevated-temperature behavior. The creep rate data differ somewhat from the rupture life data in that the lines are more parallel and have a discernible slope at all temperatures above 500 F. Since measurable extension under a steady applied load is an indication of elevated-temperature behavior, it seems apparent that such behavior becomes predominant in this alloy between 500 and 650 F. This observation is in reasonable agreement with the concept that elevated-temperature behavior commences at 0.35 to 0.50 of the absolute melting point (9) (650 F $\approx$ 0.46 melting point).

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[6] Recommended Practice for Short-Time Elevated-Temperature Tension Tests of Materials (E 21), 1961 Book of ASTM Standards, Part 3.

The plot of hardness versus temperature of Fig. 2 is similar to that found for most metals (10). It can be seen from this figure and Table 3 that the hardness of this alloy drops by more than 50 per cent (from 79.0 to 36.3 VHN) as the temperature goes from 400 to 1200 F, with a rapid drop above 900 F. These data indicate a marked reduction in tensile strength of this alloy at temperatures above 900 F. This is confirmed by the tensile data in Table 4 and Fig. 3. If the rapid drop in hardness and tensile strength is taken as a criterion for elevated-temperature behavior, it is seen that these short-time tests overestimate the true high-temperature region by nearly 400 F.

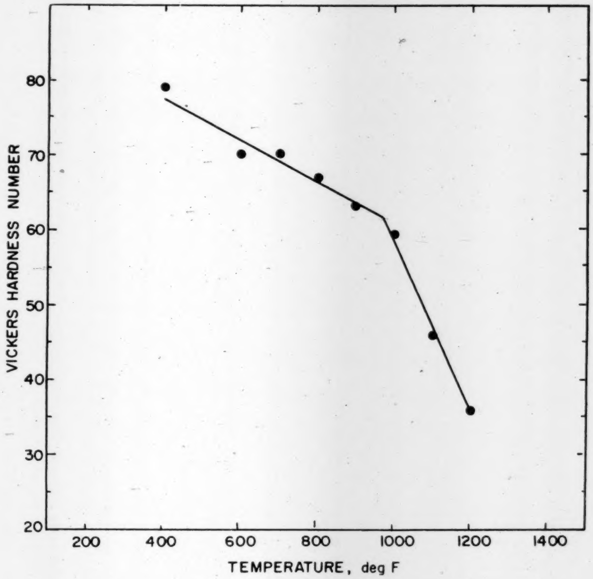


FIG. 2.—Hardness Versus Test Temperature.

TABLE 4.—TENSION TEST RESULTS AT TEMPERATURE.
Strain Rate 0.1 in. per in. per min.

Temperature, deg F	Ultimate Tensile Strength, psi	Yield Strength, (0.5 per cent Plastic Strain) psi	Elongation in 2 in. per, cent	Reduction of Area, per cent
82....	64 100	32 000	34.5	49.1
500....	53 300	26 300	31.5	46.4
650....	49 200	25 000	27.0	46.2
800....	46 700	22 500	25.6	26.6
950....	37 250	21 500	16.2	15.8
1100....	26 550	20 000	12.3	6.5

Simmons et al (6) recently examined the elevated-temperature mechanical properties of a drawn-and-stress-relieved alloy corresponding to ASTM Specifications B 122. They determined the maximum allowable stress for their alloy based on the ASME Boiler and Pressure Vessel Code by using tensile data and extrapolated creep-rupture data. It was found that the allowable stress was given by (1) one-fourth of the tensile strength (adjusted to minimum) up to 750 F; and (2) the stress for a minimum creep rate of 0.00001 per cent per hr above 750 F. The hardness and tensile data (hardness is related to tensile strength (10,11)) of the present investigation suggest that 70-30 copper-nickel in the annealed condition will also be governed by the same criteria with a transition from use of tensile strength to use of rupture data at about 800 F. This would of course be expected from the fact that the chemical compositions of the present alloy and that used by Simmons et al (6) are essentially the same.

The Larson-Miller extrapolation parameter (12) (with $C = 20$) affords a convenient means for comparison of the available data on annealed 70-30 copper-nickel. Data from the present study are plotted in Fig. 4 with the results of reference (4). The creep data of references (1–3) were widely scattered and are not included. These latter data lay, in all cases, substantially below the curves of Fig. 4(*b*).

The rupture life behavior shown in Fig. 4(*a*) indicates that the alloy of the present study is stronger than that of reference (4) at lower temperatures but weaker at higher temperatures. Creep behavior is similar. The reason for the distinct difference in strength level between the alloy of references (1–3) and those of Fig. 4 probably lies in the composition differences between them, notably the low iron content of the former alloy. (The iron causes high-temperature strengthening in 70-30 copper-nickel.) The strength differences between alloys of the same iron content

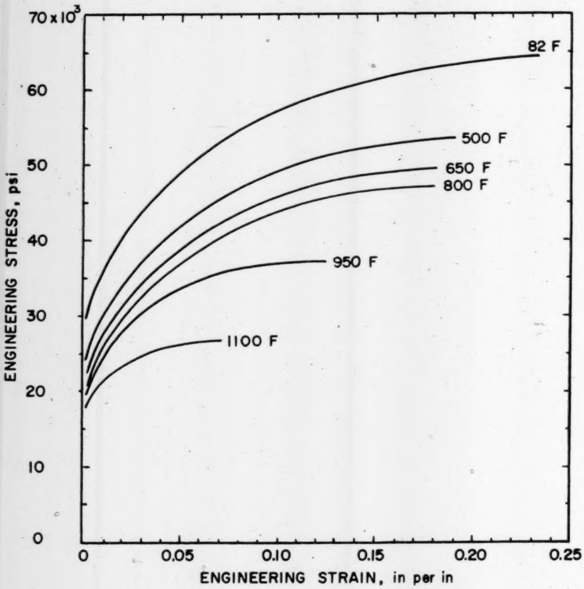
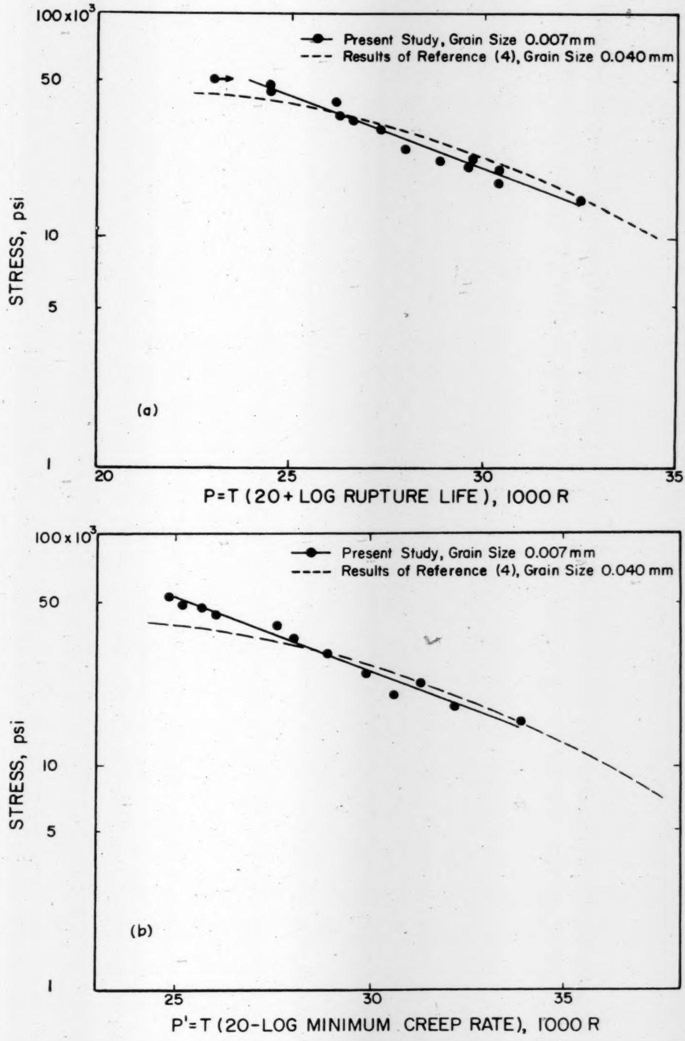


FIG. 3.—Engineering Stress-Strain Curves.

(those in Fig. 4) may be due to slight compositional variations, grain size differences, or other effects of prior thermal and mechanical treatment. ASTM Specifications B 122 permits composition limits broad enough to include all three 70-30 copper-nickel alloys for which creep-rupture data are available. Consequently a wide range of allowable stress limits may be possible due to composition fluctuations alone, without the additional complexities of thermal and mechanical treatments. In view of these facts, it would seem that the specification for 70-30 copper-nickel alloys, particularly for elevated-tempera-



(*a*) Stress versus rupture life and temperature.
(*b*) Stress versus minimum creep rate and temperature.

FIG. 4.—Larson-Miller Plots of Available Data and Present Study.

ture service, should be tightened to minimize the spread in mechanical properties.

SUMMARY

The creep-rupture, hardness, and tensile behavior of a mill-annealed 70-30 copper-nickel rod have been investigated over a range of temperatures above about 500 F. The results confirm that this annealed material has usable strength above the usual range of 300 to 500 F. The data also suggest that composition limits of ASTM Specifications B 122 should be tightened.

Acknowledgment:

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EFFECT OF COMPOSITION AND DISPERSED-PHASE PARTICLE-SIZE DISTRIBUTION ON THE STATIC ELASTIC MODULI OF TUNGSTEN-COPPER COMPOSITE MATERIALS

By Richard H. Krock[1]

Synopsis

Two-phase tungsten-copper composite materials of various compositions and dispersed-phase particle-size distributions were prepared by infiltrating a porous tungsten skeleton with copper at 1150 C in a hydrogen atmosphere. Measurements of the static elastic moduli were made with resistance strain gages. The experimentally obtained values of the elastic moduli were compared with two existing theories which predict upper and lower bounds of the modulus of a two-phase system. It was found that the elastic moduli values fell within the bounds of Paul (**6**); however, the modulus was not a single valued function of the composition but varied also with the dispersed-phase particle-size distribution.

The ability to predict the elastic and plastic properties of two-phase composite materials from a knowledge of the behavior of their constituents is a subject of widespread theoretical and practical interest. At present, little theoretical work has been done on the plastic properties of two-phase composite materials although appreciable experimental data, mostly on cermet materials (**1–4**),[2] do exist. Experiments have shown that in cases where the hard phase is nondeformable, all the plastic properties vary with the mean free matrix separation between dispersed particles (**1–3**); on the other hand, in cases where the hard phase is deformable (**5**) under the loading conditions, the plastic properties of the composite are independent of both composition and mean free matrix path and depend only on the deformation characteristics of the harder phase. In these investigations, the weaker matrix phase has been found to be strengthened by a hydrostatic component of stress.

Much theoretical work, however, has been conducted on determining the elastic moduli and some physical properties of two-phase composite materials. Paul (**6**) and Hashin and Schtrikman

The work that Dr. Krock describes in this paper was partially supported by a $6500 ASTM Doctoral Fellowship for 1961–1962. Each year ASTM awards a fellowship which provides $5000 for the fellow and $1500 for the institute. Dr. Krock was the second recipient of this fellowship. He received his doctorate in June, 1962.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

(**7**) have established upper and lower bounds for the elastic moduli of two-phase composites through use of the principles of energy and polarization-tensor minimization. These theories predict that the static elastic modulus of a two-phase composite is determined solely by the composition of the constituents. No systematic experimental investigations have been conducted to substantiate these theories. Brown (**8**) has found that the magnetic permeability of a two-phase isotropic material is not solely determined by composition but depends on the statistics of phase distribution; he suggests that other physical properties such as electrical conductivity and dielectric properties should behave similarly.

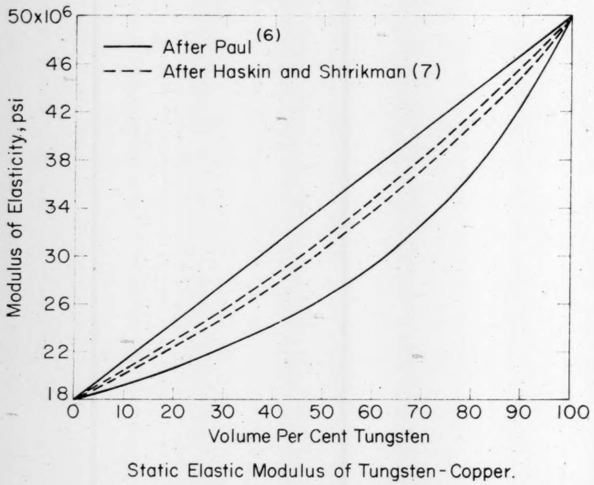


FIG. 1.—Theoretical Values of Elastic Moduli Bounds.

The present investigation was undertaken to determine whether the bounds of Paul (**6**) and Hashin and Schtrikman (**7**) would be able to predict the elastic modulus of a two-phase composite and whether the statistics of phase distribution affected the moduli of these composites.

THEORY

The two principal theories concerning the effect of microstructure on the elastic moduli of two-phase composite materials are the minimum energy theorem of Paul (**6**) and minimum polarization theorem of Hashin and Schtrikman (**7**).

Hashin has derived the bounds on the elastic constants of a two-phase material through the introduction of the elastic polarization tensor P_{ij}, defined in the following way:

If $\sigma_{ij}^{(o)}$ and $E_{ij}^{(o)}$ are the stress and strain tensor components in a deformed body of volume V and surface S, then:

$$\sigma_{ij}^{o} = \lambda_o E_{KK}^{o}\delta_{ij} + 2G_o E_{ij}^{o} = \lambda_o E_{ij}^{o}$$

where:

λ_o = Lamé constant,
G_o = shear modulus,
δ_{ij} = Kronecker delta, and
E = elastic modulus.

If a portion of this body is then replaced by a material of different modulus and the surface displacements are held fixed, the stress changes to

σ_{ij}, where $\sigma_{ij} = L_o E_{ij} + P_{ij}$, and P_{ij} = polarization tensor.

Two cases are considered to determine the upper and lower bounds on the elastic moduli of the composite, which can be regarded as being the stronger material weakened through introduction of the weaker second phase (para-elastic) and the weak phase being strengthened by additions of a stronger second phase (dia-elastic). Through minimization of the stored energy with respect to the polarization tensor, Hashin and Schtrikman (**7**) obtain upper and lower bounds for both the shear modulus, *G*, and the bulk modulus, *K*. Substituting these values into the equation relating the three elastic constants of an isotropic material,

$$E = \frac{9KG}{3K + G} \quad \ldots\ldots\ldots\ldots (1)$$

the following relationships are obtained:

Lower Bound:

$$E_1^* = \frac{9\left(K_1 + \dfrac{V_2}{\dfrac{1}{K_2 - K_1} + \dfrac{3V_1}{3K_1 + 4G_1}}\right)\left(G_1 + \dfrac{V_2}{\dfrac{1}{G_1 - G_2} + \dfrac{6}{5}\dfrac{(K_1 + 2G_1)V_1}{(3K_1 + 4G_1)G_1}}\right)}{3\left(K_2 + \dfrac{V_2}{\dfrac{1}{K_2 - K_1} + \dfrac{3V_1}{3K_1 + 4G_1}}\right) + \left(G_1 + \dfrac{V_2}{\dfrac{1}{G_2 - G_1} + \dfrac{6}{5}\dfrac{(K_1 + 2G_1)V_1}{(3K_1 + 4G_1)G_1}}\right)} \quad \ldots (2)$$

Upper Bound:

$$E_2^* = \frac{9\left(K_2 + \dfrac{V_1}{\dfrac{1}{K_1 - K_2} + \dfrac{3V_2}{3K_2 + 4G_2}}\right)\left(G_2 + \dfrac{V_1}{\dfrac{1}{G_1 - G_2} + \dfrac{6}{5}\dfrac{(K_2 + 2G_2)V_2}{(3K_2 + 4G_2)G_2}}\right)}{3\left(K_2 + \dfrac{V_1}{\dfrac{1}{K_1 - K_2} + \dfrac{3V_2}{3K_2 + 4G_2}}\right) + \left(G_2 + \dfrac{V_1}{\dfrac{1}{G_1 - G_2} + \dfrac{6}{5}\dfrac{(K_2 + 2G_2)V_2}{(3K_2 + 4G_2)G_2}}\right)} \quad \ldots (3)$$

where:

E_1^* = lower bound value of the elastic modulus of a two-phase composite consisting of materials 1 and 2, where the modulus of material 2 is greater than that of material 1,

E_2^* = upper bound value of the elastic modulus and so forth,

V_1 = volume fraction of material 1,

V_2 = volume fraction of material 2,

K_1 = bulk modulus of material 1,

K_2 = bulk modulus of material 2,

G_1 = shear modulus of material 1, and

G_2 = shear modulus of material 2.

A plot of these bounds for tungsten-copper composites (Fig. 1) shows that the upper and lower bounds are quite close, differing by about 1 per cent and varying only with composition.

Paul's bounds (**6**) on the elastic moduli of a two-phase composite are based on the minimization of strain energy and complementary energy. The derivation does not involve the polarization tensor and depends on the equivalence of stress and strain within the region of analysis.

The bounds are:

Upper Bound:

$$E_1^* = V_1E_1 + V_2E_2 \quad \ldots\ldots\ldots (4)$$

Lower Bound:

$$E_2^* = \frac{E_1 \cdot E_2}{V_2E_1 + V_1E_2} \quad \ldots\ldots\ldots (5)$$

These are identical to the bounds obtained by a simple analogy of iso-stress and iso-strain behavior between the two materials of the composite and are the common bounds obtained less rigorously by using electrical analogs. These bounds are plotted in Fig. 1 for tungsten-copper composites using the elastic constants of Table I. As can be seen, a considerably larger difference, about 20 per cent, exists between the Paul bounds than between the Hashin bounds, but the only variable in Eqs 4 and 5 is the volume fraction.

TABLE I.—ELASTIC CONSTANTS OF TUNGSTEN AND COPPER USED FOR THE CALCULATION OF ELASTIC MODULI BOUNDS.[a]

	Poisson's Ratio	Bulk Modulus, K, psi	Shear Modulus, G, psi	Young's Modulus, E, psi
Tungsten	0.30	41.66×10^6	19.23×10^6	50×10^6
Copper	0.35	20.0×10^6	6.7×10^6	18.0×10^6

[a] C. J. Smithells, *Metals Reference Book*, Vol. 2, Butterworth Press, Washington, D. C. (1952), p. 614.

TABLE II.—SIZE DISTRIBUTION OF TUNGSTEN POWDER.[a]

A	0.5	1	2	3	4	5	6	7	8	9	10
	0.01	0.20	6.54	16.58	19.48	18.67	15.19	10.62	5.83	4.15	2.73
B	150 to 105	105 to 74	74 to 53	53 to 44	44 to 38	..38					
	...	...	0.01	0.017	0.054	0.919					
C	<38 μ part of B—no details of statistics known										
D	0.028	0.135	0.137	0.114	0.188	0.396					
E	0.02	0.03	0.04	0.06	0.14	0.71					
F	...	0.06	0.22	0.28	0.25	0.19					
G	<38 μ part of F—no details of statistics known										

[a] Upper row of figures are diameters in microns; lower row are per cent distributions.

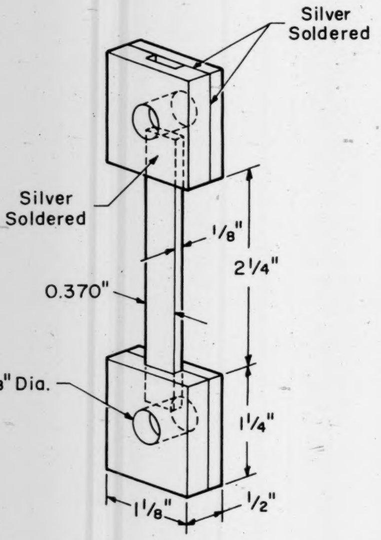
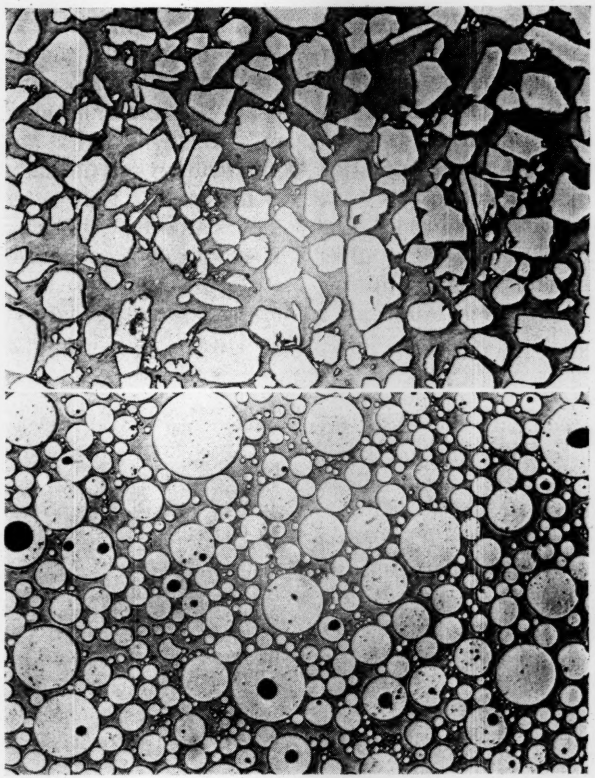


FIG. 2.—Specimen and Gripping Arrangement.

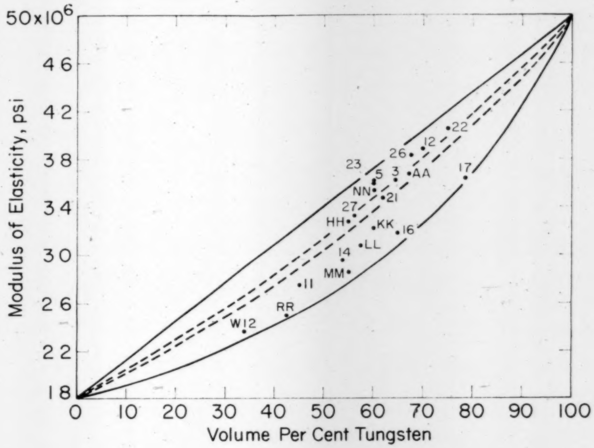
EXPERIMENTAL PROCEDURE

Two-phase composites of tungsten and copper were prepared by powder metallurgy techniques. Seven size distributions of tungsten powder (Table II) were coated with poly(ethylene glycol) and pressed in a steel die into 4 by $\frac{1}{2}$ by $\frac{1}{8}$-in. compacts at several pressures to vary the green density. A weight of copper sufficient to fill the void volume was infiltrated into the tungsten compact. The heat treating was carried out in an atmosphere of prepurified dry hydrogen and consisted of slow heating (150 C per hr to 600 C) to prevent compact fracture from evolution of the poly(ethylene glycol) binder, an iso-



(*Top*) Specimen No. 21, E tungsten powder (61.85 per cent tungsten by volume).
(*Bottom*) Specimen No. 26, Linde microspheres (D) (67.50 per cent tungsten by volume).

FIG. 3.—Photomicrograph of Tungsten-Copper Composite (×160, unetched).



Static Elastic Moduli of Tungsten-Copper

FIG. 4.—Experimental Data Obtained on Tungsten-Copper Composites.

thermal hold at 900 C for 1 hr to remove any surface oxidation on the tungsten, followed by infiltration of the copper at 1150 C. The compact was held at the infiltration temperature for 15 min and then furnace cooled to room temperature in 3 hr. The infiltrated compacts were machined into rectangular blocks, and steel pin grips (Fig. 2) were silver soldered to them for tension testing. Tension testing was carried out at a crosshead motion of 0.002 in. per min. SR-4 strain gages were mounted on both sides of the specimen and the output was fed into an *X*-*Y* recorder. The strain sensitivity was 2.0 μin. per in. The volume fractions of the two phases were determined by quantitative metallography using standard lineal analysis. The microstructure of the composites consisted of dispersed tungsten particles in a copper matrix (Fig. 3).

TABLE III.—COMPILATION OF DATA FOR TUNGSTEN-COPPER COMPOSITES.

Specimen No.	Particle Size Distribution	Composition (Volume per cent tungsten)	Mean-free Matrix Path	Static Room-Temperature Elastic Modulus, psi
3	E	64.35	1.03	36.25×10^6
5	A	60.00	1.78	36.25
11	G	45.00	1.30	27.50
12	B	70.00	2.80	38.75
W12	C	33.75	1.95	23.50
14	G	55.80	2.12	29.50
16	A	65.00	1.38	31.75
17	C	78.75	0.907	35.63
21	E	61.85	21.30	34.75
22	(D)	75.00	20.90	40.50
23	(D)	60.00	19.70	35.75
26	(D)	67.50	14.95	38.25
27	(D)	56.25	23.70	33.25
AA	(D)	66.85	12.50	36.75
HH	F	55.00	15.30	32.25
KK	B	60.00	3.50	32.75
LL	C	60.00	2.50	30.75
MM	C	55.00	3.06	28.50
NN	G	60.00	2.50	35.25
RR	C	42.50	2.70	25.00

RESULTS AND DISCUSSIONS

Using the values of the elastic constants given in Table I, the upper and lower bounds for the modulus of tungsten-copper composites were computed using the minimum energy relationships of Paul (Eqs 4 and 5) and the variational polarization relationships of Hashin and Schtrikman (Eqs 2 and 3, Fig. 1). The variational polarization bounds of Hashin and Schtrikman are much more stringent than those of Paul; the bounds are separated by only about 1 per cent. Figure 4 shows the same plot of the moduli bounds with the experimental data superimposed. The specimen numbers included in the plot are those described in Table III.

As can be seen, the experimental elastic moduli lie within the theoretical bounds set by Paul but outside the bounds of Hashin and Schtrikman, although the proximity to the Hashin bounds increases with increasing volume fraction. The Paul bounds represent the extremes between the constituents of the composite deforming under equal stress or equal strain and therefore must encompass all values. The variational polarization bounds of Hashin and Schtrikman do not describe the moduli of these composites at low volume fractions (65 volume per cent copper). Both the theoretical derivations predict a single-valued modulus depending only on the volume fraction of the phases in the composite and do not account for statistical deviations about this volume fraction resulting from a distribution of particle sizes and mean interparticle separations.

Figure 4 indicates that at any value of composition the rounded particles exhibited a higher elastic modulus than the angular hydrogen-reduced powders.

For the tungsten-copper composites, the elastic modulus is a function of these statistical variations resulting from particle-size distributions, and although the modulus does fall at a given volume fraction within the Paul bounds (1), the absolute value is a function of particle-size distribution, as can be seen from the vertical constant-volume intercepts of Fig. 4. The elastic modulus of these composites, however, does not depend on the experimentally determined mean-free path, as observed in plastic properties of cermets, in any regular way but upon the actual distribution of particle sizes and spacings. The mean-free matrix path has been shown to affect the yield strength (2–4), tensile strength, and impact strength of cermets; but no such effect has been reported to date on the elastic modulus. Other physical properties affected by such statistical distributions are the magnetic permeability, electrical conductivity, and dielectric strength as shown by Brown (8).

SUMMARY AND CONCLUSIONS

The static elastic modulus of two-phase tungsten-copper composites was investigated as a function of composition, average particle size, and particle-size distribution. Two theoretical expressions based on energy and variational polarization vector minimization have provided bounds for the elastic modulus of two-phase systems as a monotonic function of volume fraction. The experimental results showed:

1. The elastic moduli of tungsten-copper composites fell within the equal-stress - equal-strain minimum energy bounds derived by Paul, but were inconsistent with the more stringent variational bounds of Hashin and Schtrikman.
2. The proximity of the experimental data to the Hashin bounds increases with increasing composition.
3. The static elastic moduli of tungsten-copper composites were not determined solely by the composition, but depended also on particle-size distribution and distribution of interparticle spacings.
4. For any given composition, specimens containing rounded particles of the dispersed phase exhibited a higher elastic modulus than specimens containing angular particles of the dispersed phase.

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STRESS-STRAIN CHARACTERISTICS OF METALS UNDER CONDITIONS OF TRANSIENT HEATING AND LOADING

By A. C. Willhelm[1] and J. R. Kattus[1]

Synopsis

An investigation was conducted to determine the stress-strain characteristics of metals under transient conditions of simultaneous heating and straining, and to determine the applicability of the mechanical equation of state as a means of predicting these stress-strain characteristics from ordinary tensile data. It is shown that, at constant strain rates, the load-carrying capacity of metals tends to decrease with increasing constant heating rates. Although the predicted transient stress-strain curves were similar to the experimental curves, there were significant and consistent differences. The discrepancies are believed to be a result of possible influences of a strain aging type reaction that is manifested at larger degrees of strain under the transient conditions than in conventional tensile determinations.

Tensile and creep data are generally used as the bases for the selection of alloys and the design of load-bearing structures for elevated-temperature service. In creep determinations both load and temperature are controlled at constant levels while the specimen deforms at a varying rate; in tensile determinations the temperature and rate of straining are controlled at approximately constant levels while the load varies continuously. These controlled conditions are not representative of certain types of elevated-temperature service. Solid-fuel rockets, for example, must sustain high, relatively constant loads while they are rapidly heated. Design data for such applications can be obtained with a special "high heating rate" test that has been developed to simulate these service conditions more closely than tension and creep tests do (**1**).[2] In this test the specimen is subjected to a constant load and is heated at a constant rate to failure, while the strain is recorded as a function of temperature.

In many applications, however, particularly aircraft and missile components, loads and temperatures vary simultaneously. Such transient conditions are not represented in any of the conventional methods for obtaining design data. Consequently an investigation was conducted to determine the short-time stress-strain characteristics of metals under transient conditions of heating and loading. Although an unlimited number of transient conditions is conceivable, this investigation was limited to the simplest type wherein heating and straining are initiated simultaneously and continued at constant rates until the specimen fails. A further purpose of the work was to evaluate the efficacy of the mechanical equation of state as a means of predicting transient stress-strain behavior from ordinary tensile stress-strain curves. Such a correlation would provide a rational basis for using conventional data in designing for transient conditions.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

MECHANICAL EQUATION OF STATE

The mechanical equation of state, first suggested by Ludwick in 1909 (2), has served as a guiding principle for many investigators seeking to formulate theoretical or empirical equations relating the flow stress of metals to its pertinent parameters. The concept of the mechanical equation of state suggests that the instantaneous flow stress in a given metal is determined entirely by the instantaneous values of strain, strain rate, and temperature and is independent of prior conditions of straining and heating. Therefore when a specific alloy is heated and loaded, any given combination of strain, strain rate, and temperature would be expected to produce the same value of stress regardless of the paths by which these three variables reached the given combination.

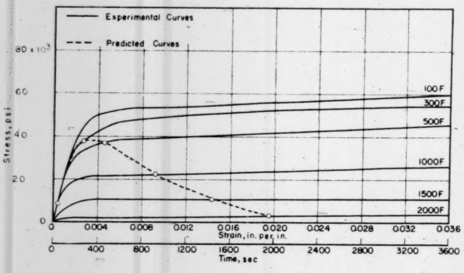


FIG. 1.—Family of Tensile Stress-Strain Curves Obtained at Constant Temperatures and at a Constant Strain Rate of 1×10^{-5} in. per in. per sec with a Graphically Constructed Curve Predicting Stress-Strain Behavior at the Same Strain Rate and a Simultaneous Heating Rate of 1.0 F per sec.

Although most experts believe that the mechanical equation of state is not valid except for restricted conditions (6–8), others contend that the available evidence either supporting or contradicting the concept is inconclusive (9). Regardless of its general validity, it is certainly possible that the equation can serve as a useful approximation for correlating mechanical-property data or that it is valid for the restricted range of short-time conditions covered in this work.

A graphic method used for predicting transient stress-strain behavior from tensile data is illustrated in Fig. 1. The solid curves in this illustration are conventional stress-strain curves obtained in tension tests on annealed Type 321 stainless steel sheet at a constant strain rate of 0.00001 per sec and at various constant temperatures from 100 to 2000 F. Since a constant strain rate was used in obtaining these curves, a time scale corresponding to the strain scale can be shown on the abscissa. These curves were used to construct the dotted curve which is a prediction of the stress-strain characteristics of the same material when loaded at the same strain rate and simultaneously heated at a rate of 1.0 F per sec. Points for the predicted transient curve were determined for each temperature represented by the conventional curves. At a transient heating rate of 1.0 F per sec and a strain rate of 0.00001 per sec, the temperature would increase from 75 F (room temperature) to 500 F, for example, in 425 sec, which corresponds to a strain of 0.00425 in. per in. These values of temperature, strain, and strain rate are identical to those existing after 425 sec on the conventional 500-F stress-strain curve. In accordance with the mechanical equation of state, then, the stress for the transient curve under these conditions would be equal to that on the conventional curve, and this point is positioned accordingly at the stress corresponding to the 425-sec intercept on the conventional stress-strain curve. Points for other transient temperatures are similarly positioned on Fig. 1 by means of the other constant-temperature stress-strain curves to form the predicted stress-strain curve for a heating rate of 1.0 F per sec. Experimentally recorded transient stress-strain curves include thermal expansion in the strain measurements whereas the conventional curves do not. For a more accurate prediction of experimental transient curves, therefore, the points forming the predicted curve should be corrected for thermal expansion by increasing their strain values by an amount equal to thermal expansion at each temperature. The same techniques can be used to construct predicted transient curves for other heating rates and strain rates.

For any particular strain rate significant transient stress-strain curves are restricted to a certain range of heating rates. If the heating is too slow in comparison with the straining, the specimen is strained to failure before appreciable heating occurs; if the heating is too fast, the specimen melts before significant straining occurs.

SCOPE OF STUDY

The experimental work was carried out on one alloy—annealed Type 321 stainless steel sheet. This material was chosen because of its availability, stability, and reasonably good high-temperature strength. The stability, of course, minimizes structural changes which might affect the stress-strain characteristics and thereby complicate the interpretation of the results.

Triplicate tensile stress-strain curves were recorded at constant temperatures of 100, 500, 1000, 1500, and 2000 F for each of three constant strain rates: 0.00001, 0.0001, and 0.001 per sec.

By means of stress-strain curves representing the averages of the three curves for each condition, predicted stress-strain curves for conditions of simultaneous heating and loading were constructed in the manner described above for the following heating rates and strain rates: (1) 0.25, 1.0, and 2.0 F per sec at a strain rate of 0.00001 per sec; (2) 2.5, 5.0, and 20 F per sec at a strain rate of 0.0001 per sec; and (3) 25, 100, and 200 F per sec at a strain rate of 0.001 per sec. Triplicate stress-strain curves which showed a surprisingly small amount of scatter, were then obtained experimentally on specimens subjected to the same transient conditions. The experimental curves and predicted curves were compared to illustrate the degree of accuracy of the method of prediction.

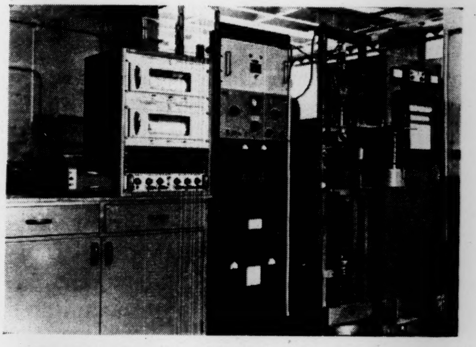


FIG. 2.—Tension Machine for Programmed Heating and Straining.

TEST EQUIPMENT

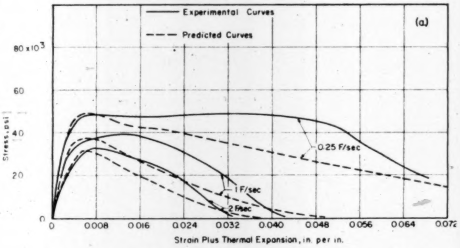
The basic equipment, shown in Fig. 2, includes a special screw-driven tension machine in which the crosshead can be driven at rates up to 6 in. per sec. The general characteristics of this machine which has been used at Southern Research Institute for many investigations of the tensile properties of metals over wide ranges of strain rate and temperature have been described in previous publications (3–5). Briefly, the crosshead is driven by a d-c motor through appropriate speed reducers. The loading frame with a specimen, clip-on extensometer, and load cell in place is shown at the right of center in Fig. 2. The specimens are resistance heated by current supplied from a specially wound transformer contained in the complete heating unit at the right-hand side of Fig. 2. Specimen heating is regulated by a servo-control circuit that includes thyratrons and a current-adjusting-type control unit as well as the transformer. This heating unit can produce rapid heating rates up to 2000 F per sec. Specimen temperature is measured by means of thermocouples flash welded to the specimen. For temperature recording and control in tensile determinations the thermocouples actuate a strip-chart recorder-controller in the servo-control circuit. Special load cells and extensometers with strain-gage transducers are used to detect load and strain. The calibrated a-c outputs from these transducers are amplified, demodulated, filtered, and then fed into a suitable recorder. With the exception of the recorder, instruments for these purposes as well as the motor control are contained in the center rack in Fig. 2. In tensile determinations stress-strain curves are recorded directly on an *x-y* recorder or oscilloscope. In the transient heating and loading determinations load, strain, and temperature are all recorded as functions of time on a multichannel oscillograph, the small instrument on the left-hand side of Fig. 2. This type of recording is desirable for the simultaneous heating and loading conditions to show whether suitable synchronization of heating and straining is achieved. The stress-strain curves for the transient conditions are constructed from corresponding stress and strain values on the oscillograph record.

Two analog programmers, shown near the left-hand side of Fig. 2, are used to obtain with reasonable precision any preselected heating and straining programs with respect to time. One of the programmers is installed in the heating-control circuit, and the other is installed in a circuit that, in simplified terms, uses the strain signal to control the motor speed in accordance with a preselected strain program. With these installations the selected constant strain rates for the tensile determinations and the combined constant heating and strain rates for the transient stress-strain determinations were readily obtained.

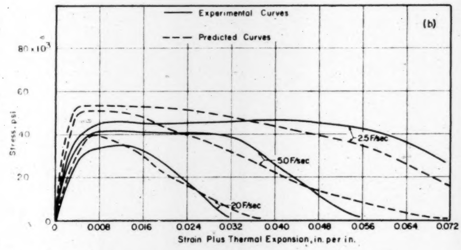
Since thermal expansion was included in the strain measurements during simultaneous heating and loading, the strain programs for these determinations included thermal expansion in addition to mechanical strain in order to obtain the desired mechanical strain rate. As mentioned previously, since the recorded transient stress-strain curves include thermal expansion, thermal expansion is added to the strain values of the predicted curves to provide valid comparisons.

EXPERIMENTAL RESULTS

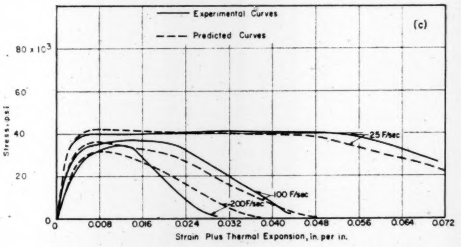
The comparisons of the predicted and experimental stress-strain curves for simultaneous heating and loading are shown in Figs. 3(*a*), (*b*), and (*c*) pertaining to strain rates of 0.00001, 0.0001, and 0.001 per sec respectively. The experimental curves represent averages of three determinations. Although they have the same general shape as the predicted curves, they are not identical. Generally, the types of deviations between the two types of curves are similar regardless of variations in strain rate and heating rate. The experimental stress values tend to fall below the predicted values at low strain levels, to rise above the predicted values at intermediate strain levels, and again to fall below the predicted values before failure. The extent of these deviations varies considerably with the different strain rates.



(*a*) Constant strain rate of 1×10^{-5} in. per in. per sec.



(*b*) Constant strain rate of 1×10^{-4} in. per in. per sec.



(*c*) Constant strain rate of 1×10^{-3} in. per in. per sec.

FIG. 3.—Family of Experimental and Predicted Stress-Strain Curves for a Constant Strain Rate and Various Simultaneous Constant Heating Rates.

At the most rapid strain rate (Fig. 3(*c*)) the deviations are quite small, the predicted and experimental curves being in close agreement. At the slowest strain rate (Fig. 3(*a*)) the deviations are somewhat larger, particularly at intermediate strain levels. The greatest deviations occur at the intermediate strain rate (Fig. 3(*b*)), which shows that the experimental stress levels fall well below the predicted levels in the initial stages of plastic deformation. At higher strain levels, on the other hand, the experimental and predicted curves are in reasonably close agreement.

In spite of the consistent discrepancies between the two types of curves, the predicted and experimental maximum stress values (transient ultimate strength) for corresponding heating rates are reasonably similar at the slowest and fastest strain rates. At the intermediate strain rate, however, the experimental transient ultimate strengths are considerably lower—as much as 10,000 psi—than the predicted values.

Both the experimental and the predicted transient stress-strain curves show that, at any given strain rate, both the load-carrying capacity and the total strain absorption of the Type 321 stainless steel decrease with increasing heating rates. These effects of heating rate under conditions of simultaneous transient heating and loading would be expected in all metals unless the metallurgical structure is extremely unstable—for example, to the extent that strength increases with temperature. Generally, total elongation measurements on the ruptured specimens were of no metallurgical significance since the specimens failed by melting rather than by mechanical rupture under most of the transient conditions investigated.

The reasons for the discrepancies between the predicted and experimental transient stress-strain curves have not been conclusively established. The possibility that the deviations represent normal data scatter is ruled out because of their consistency among triplicate sets of specimens and among different experimental conditions. Although the programming of heating rates and strain rates was accomplished with the degree of precision that can be expected with commercial components, minor variations from the desired rates did occur in the experimental work. It is possible that these variations contributed to the discrepancies between the two types of curves. This source of error, however, would be expected to result in random scatter in the data rather than in the consistent discrepancies that occurred.

It is more probable that the discrepancies are caused by a certain amount of structural instability in the Type 321 stainless steel test material which had not been anticipated at the start. Certain peculiarities in the conventional tensile data indicate that a strain-temperature-time-dependent structural change, which had a strengthening effect, occurred over a temperature range up to 1000 F. This type of structural change, which was manifested by minor serrations in some stress-strain curves and by reversals in the normal strengthening effects of increased strain rate, is analogous to strain aging (10). In the tensile determinations at constant temperatures, the strengthening effects of this instability became pronounced in the early stages of yielding. In the transient tensile determinations, apparently the strengthening effects were delayed until larger amounts of strain had occurred. This delay in strain-age strengthening probably accounts for the fact that the experimental transient stress values were below the values predicted from ordinary tensile data at low strain levels and above the predicted values at higher strain levels. The lag in the strain level for strain-age strengthening in the transient tests is consistent with the fact that the driving force for this type of reaction involves both strain and temperature. When a specimen is strained to any particular combination of strain and temperature, the temperature during that straining period in simultaneous heating and loading lags behind that in the constant-temperature tensile determinations. For equivalent driving force for the strain-aging reaction, then, the transient specimens must be strained more than the conventional tension specimens.

The large discrepancies between the experimental and the predicted transient stress-strain curves at the intermediate strain rate as compared with those at the other strain rates are probably associated with the fact that the greatest manifestations of a strain aging type reaction, particularly increased flow stress in the initial stages of yielding, occurred in the conventional tensile determinations at this strain rate.

The discrepancies between the experimental and predicted curves and their possible association with a temperature-strain-time-dependent structural change are indicative that, contrary to the mechanical equation of state, instantaneous flow stress in certain alloys is dependent to some extent on the prior conditions of straining and heating as well as upon the instantaneous values of strain, strain rate, and temperature.

SUMMARY

Under simple transient conditions of heating and loading, wherein heating and straining proceed at constant rates, the load-carrying capacity and total strain absorption of metals tend to decrease with increasing heating rates. By means of the mechanical equation of state, the stress-strain characteristics of metals under such conditions can be predicted approximately from conventional tensile stress-strain curves obtained at various constant temperatures and at strain rates equal to those applied under transient conditions. Similar techniques could probably be used to make approximate predictions of stress-strain characteristics under more complicated transient conditions. Although more conclusive evidence is needed, the data indicate that inaccuracies in the predictions can be caused by possible effects on flow stress of temperature- and strain-dependent structural instabilities which are not taken into account in the mechanical equation of state. Specifically, in the Type 321 stainless steel used in this work a strain aging type reaction, which is manifested at higher strain levels under simultaneous heating and loading conditions than in conventional tensile determinations, is believed to be the main cause of the discrepancies between the experimental and predicted transient stress-strain curves.

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THERMAL EXPANSION OF FIVE TITANIUM CARBIDE CERMETS FROM 68 TO 1800 F*

By L. C. Harrington[1] and G. H. Rowe[1]

Synopsis

This paper presents measurements of the thermal expansion of five titanium carbide type cermets from 68 to 1800 F. These cermets are designated by Kennametal, Inc., as K 138A, K 150A, K 151A, K 152B, and K 162B. They contain from 64 to 80 weight per cent titanium carbide, 10 to 30 weight per cent metal binder (cobalt, nickel, or nickel and molybdenum), and 6 to 10 weight per cent other carbides.

An attempt was made to calculate the thermal expansion of each type cermet from the thermal expansions of the constituents. The expansion of the mixture was computed by weighting the expansions of the constituents according to (1) weight per cent of the constituents, (2) volume per cent of the constituents, and (3) according to a value developed for mixtures by P. S. Turner (1).[2] Expansions computed according to volume per cent and by Turner's method agreed with measured values within ±5 per cent, while the values calculated by weight per cent were from 5 to 11 per cent higher than the observed values.

The thermal expansions of these cermets are compared with the expansions of a group of metals and alloys.

Measurement of the thermal expansion of five titanium carbide cermets was undertaken to evaluate materials for valves and bearings at elevated temperatures. An attempt was made to find a method for calculating the thermal expansion of such mixtures.

Specimens

The materials studied were Kennametal, Inc. catalogue mixtures K 138A, K 150A, K 151A, K 152B, and K 162B with nominal compositions shown in Table I.

The dilatometer specimens were approximately 0.158 in. (4 mm) in diameter and 1.97 in. (50 mm) long. The measured bulk density of each material is shown in Table I, together with nominal and calculated density values.

Method

Tests:

The bulk densities of these specimens were obtained by dividing the weight by the volume. The specimens were weighed on an analytical balance to the nearest 0.0001 g. The length and diameter were

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[1] Materials Laboratory-CANEL, Pratt and Whitney Aircraft, Division of United Aircraft Corp., Middletown, Conn.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

TABLE I.—CHEMICAL COMPOSITION AND DENSITY.

	Nominal Chemical Composition, weight per cent					Density, g per cu cm		
	Nickel	Cobalt	Molybdenum	Cb(TaTi)C[a]	TiC	Measured	Calculated	Nominal (Kennametal)
K 150A.........	10	...	...	10	80	5.82	5.34	5.8
K 151A.........	20	...	...	10	70	5.64	5.61	5.8
K 138A.........	...	20	...	10	70	5.75	5.61	5.8
K 162B.........	25	...	5	6	64	6.07	5.84	6.0
K 152B.........	30	...	...	6	64	5.93	5.81	6.0

[a] Added as a solid solution of CbC, TaC and TiC, containing about 90 per cent CbC.

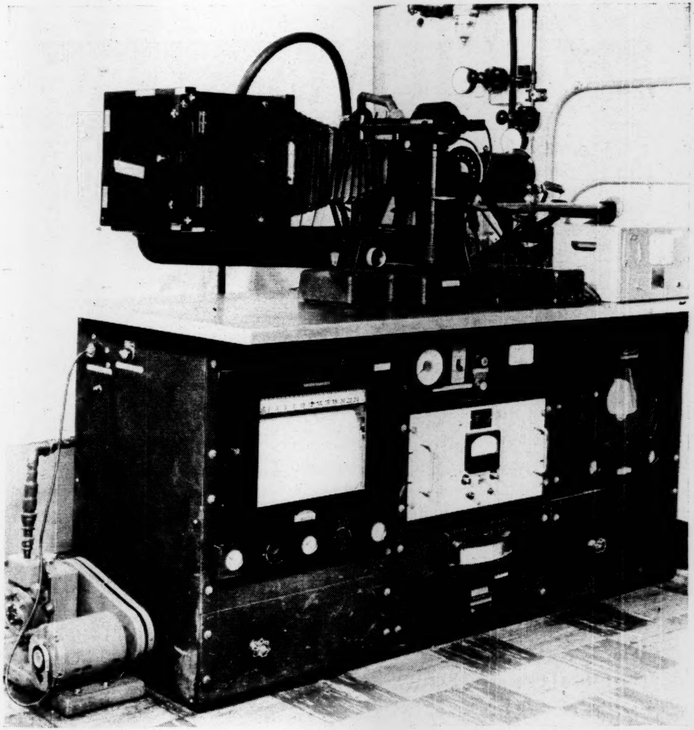

FIG. 1.—Thermal Expansion Apparatus.

measured with micrometer calipers to the nearest 0.0001 in.

The thermal-expansion measurements were made with the aid of a Leitz dilatometer (Figs. 1 and 2) in which the specimen expansion is magnified about 200 × and recorded on a photographic film by a beam of light. A change in length of 0.1 mm may be estimated on the photographic film which represents a change in specimen length of 0.0005 mm (0.00002 in.). For a specimen 50 mm

(1.97 in.) long, this represents a change in length of 10^{-5} mm per mm or in. per in. The reproducibility of the instrument, at the heating rate used in this study on a standard material such as copper, has been established as within ±3 per cent. The dilatometer chosen for use in our laboratory is described in ASTM Recommended Practice E 80[3] as the rotating mirror method. Its approximate accuracy as given in E 80 is 1×10^{-5} in. In addition to accuracy, this dilatometer also had provision for heating the specimen in either a vacuum or an inert atmosphere. The expansion record made with this dilatometer is continuous, so that it may be used to measure the temperature at which a crystal transformation occurs in such metals as iron. Other dilatometers listed in ASTM Methods C 372[4] and B 95[5] have a precision of 1×10^{-4}, and the expansion record is discontinuous.

The magnification factor of the instrument, which is calibrated annually, is determined by measuring the distance between dots made on the photographic film for each 0.2-mm change in specimen length. The magnification factor (196.2) has not changed more than 0.3 per cent in the last five years.

For recording the expansion results, a film was selected that shrinks less than 0.5 per cent during processing. The films were exposed, stored, developed, and measured at the same constant temperature and humidity, which minimized dimensional changes in the film.

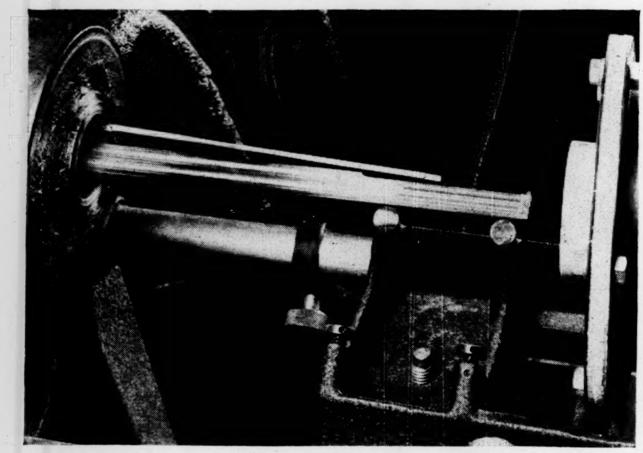

FIG. 2.—Thermal Expansion Apparatus Showing Specimen in Place.

Heating of the specimen was accomplished by a program-controlled electric resistance furnace. At the lower temperatures, heating was supplied at the rate of 3 F per min and was gradually increased to 5 F per min. Heating from room temperature to 1800 F was accomplished in 6½ hr. When a constant heating rate of 4 F per min was used, the results showed a lag in specimen expansion at temperatures below 800 F. When the heating rate from room temperature to 800 F was reduced, this lag was avoided.

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[3] Recommended Practice for Dilatometric Analysis of Metallic Materials (E 80), 1961 Book of ASTM Standards, Part 3.

[4] Method of Test for Linear Thermal Expansion of Fired Whiteware Products by the Dilatometer Method (C 372), 1961 Book of ASTM Standards, Part 5.

[5] Method of Test for Linear Expansion of Metals (B 95), 1961 Book of ASTM Standards, Part 3.

The specimens were heated in a vacuum at a pressure of from 0.1 to 0.4 μ. This vacuum was probably partially responsible for the poor heat transfer to the specimen at low temperatures.

The temperature of the cermet test specimen was assumed to be equal to the temperature measured with a chromel-alumel thermocouple, whose hot-junction was located in the groove at the center of a standard chronin (90 Ni, 10 Cr) specimen. The test specimen and standard specimen were adjacent and symmetrically placed in the test zone (Fig. 2). Temperatures were determined from a recorder graduated in 10 F intervals. The assumption of temperature was justified by comparing temperature measurements made at the center and both ends of a test specimen with the standard chronin specimen temperature shown on the indicator recorder. The mean specimen temperature agreed with recorder within 4 F at 1800 F. The maximum variation between the indications of the three thermocouples was 9 F at 900 F and 20 F at 1800 F. The three thermocouples were tack welded to the test specimen. This temperature variation was considered acceptable within the limits of measurement accuracy. The test specimen and a standard specimen were placed horizontally in quartz troughs. The change in length of each was transmitted by a quartz rod to the head of the instrument. As these specimens expanded, the quartz rods changed the angle of the prism from which the beam of light was reflected to the photographic film. Thus, the change in specimen length caused a line to be drawn across the photographic film. The test specimen displaced the light vertically while the standard displaced it horizontally. If the test specimen and the standard specimen are of the same material, the trace on the film would be a straight line at an angle of 45 deg. During heating, the light was turned off at 100 F intervals. When analyzing the film, the vertical displacement of the light from the starting point was read at the breaks in the line. An expansion measurement was thus obtained for each 100 F. These readings were in millimeters on the film and had to be divided by the magnification factor and the specimen length to bring the results to units of inches per inch of specimen length. A correction was made for the expansion of the 2-in. length of quartz trough under the specimen. No correction is necessary for the remaining length of trough and push rod since their expansions compensate for one another.

A parabola, $y = A + BT + CT^2$, was fitted to the temperature-expansion data by the least squares method. Two or more runs were made for each material and the curve fitted the experimental data within ± 3 per cent for all temperatures above 800 F. The mean coefficient, α, from 68 F to T is $y/(T - 68)$. The instantaneous coefficient of expansion is the slope of the expansion-temperature curve, or, $dy/dT = B + 2CT$.

Calculations:

An attempt was made to find a method for calculating the thermal expansion of these cermets from the known expansions of the constituents. The method presented by Turner (1)[2] seemed the best approach for our material. He considered the coefficient of expansion of a mechanical mixture as the result of the stresses set up by the difference in coefficient of expansion of the constituents. The re-

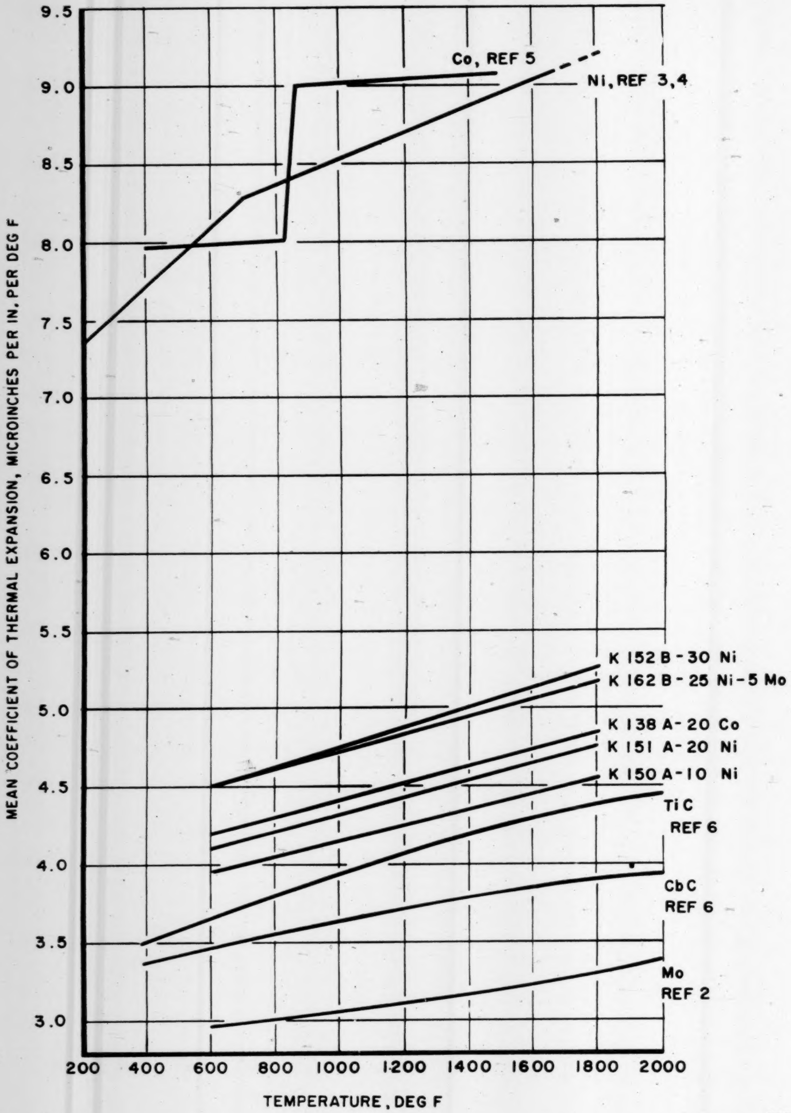

FIG. 3.—Mean Coefficients of Thermal Expansion of Five Titanium Carbide Cermets and of Their Constituents (Room Temperature to Temperature Deg Fahr).

sulting coefficient of expansion would be:

$$\alpha_r = \frac{\frac{\alpha_1 P_1 K_1}{d_1} + \frac{\alpha_2 P_2 K_2}{d_2} + \cdots + \frac{\alpha_n P_n K_n}{d_n}}{\frac{P_1 K_1}{d_1} + \frac{P_2 K_2}{d_2} + \cdots + \frac{P_n K_n}{d_n}}$$

where:

α = mean coefficient of thermal expansion,
P = weight fraction of constituent,
K = bulk modulus, and
d = density.

Subscripts *i* (1 to *n*) and *r* refer to the *i*th component and to the resultant mixture, respectively. It can be seen that if the *K*'s in the above formulas are nearly equal, the calculated α will be proportional to the volume fraction of the constituents. If the *K*:*d* ratios are nearly equal the resulting α will be nearly proportional to the weight fractions of the constituents. For the constituents in this study, the *K*'s are about equal with the exception of molybdenum. The *K*:*d*

TABLE II.—VALUES FOR THE BULK MODULUS AND DENSITY USED IN THESE CALCULATIONS.

	Nickel	Cobalt	Molybdenum	Cb(TaTi)C	TiC
K, 10^6 psi	26.3	25	43.6	24	25
d:					
g per cu cm	8.90	8.90	10.24	7.72	4.90
lb per cu in.	0.322	0.322	0.369	0.279	0.177
K/*d*, 10^6 in.	81.7	77.6	118.2	86.0	141.2

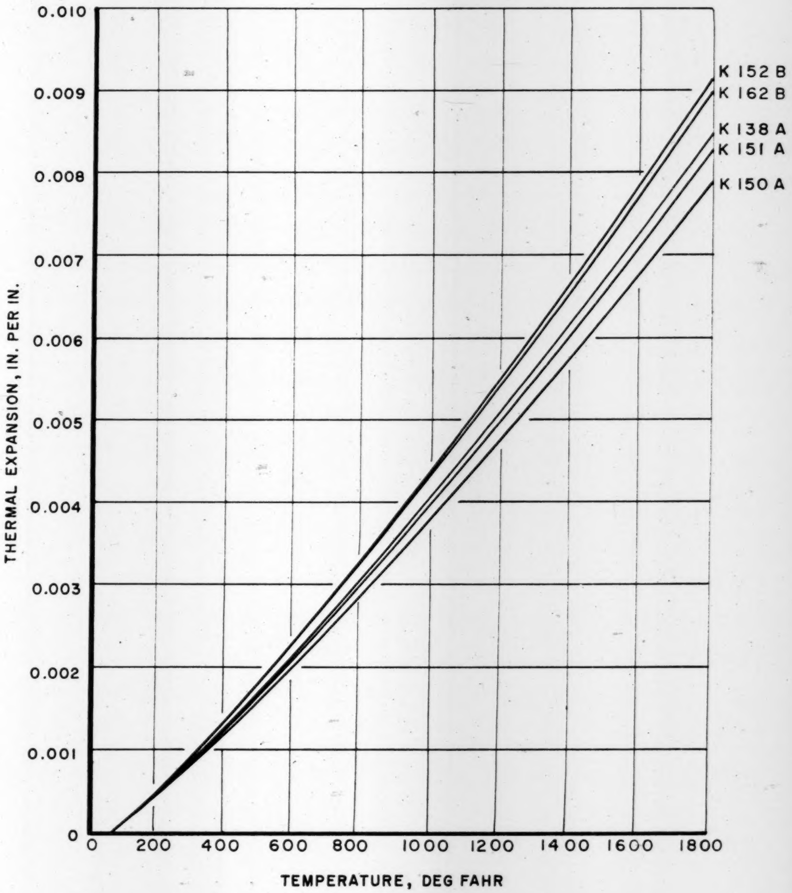

FIG. 4.—Thermal Expansion of Five Titanium Carbide Cermets.

ratio for TiC is about twice that of nickel or cobalt.

In calculating the thermal expansions of these cermets, the mean coefficients of thermal expansion of the constituents used are shown in Fig. 3. The values for the bulk modulus and density of nickel, cobalt, molybdenum, columbium-tantalum-titanium mixed carbides, and titanium carbide used in our calculations are given in Table II. The thermal expansion of molybdenum was measured in our laboratory (2). The expansion data for nickel from room temperature to 1652 F and from 1000 to 1800 F are recorded in references (3) and (4), respec-

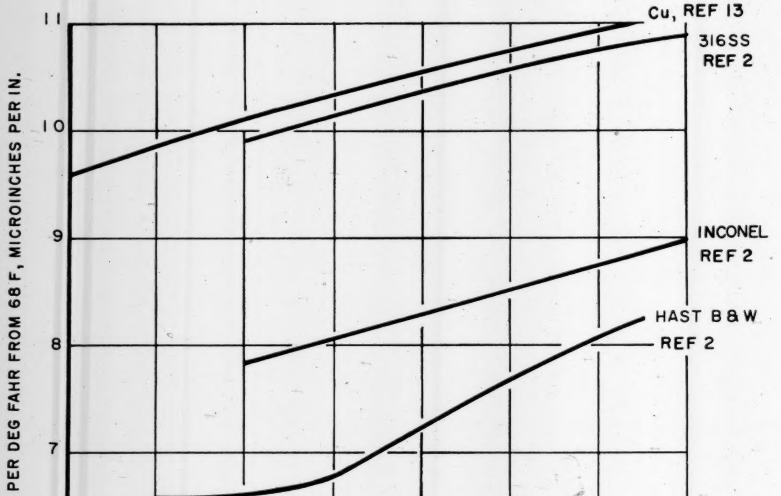

FIG. 5.—Comparison of the Thermal Expansions of Some Metals and Alloys with the Expansions of These Five Titanium Carbide Cermets. (Mean coefficients room temperature to temperature deg Fahr.)

tively. The thermal expansion data for cobalt were from measurements by Fine and Ellis (5). The expansion data for the carbides were measurements by Mauer and Bolz (6). These measurements were made by the X-ray diffraction method on a single crystal. Expansion measurements made on a specimen rather than on a single crystal would be more representative of the material in this study.

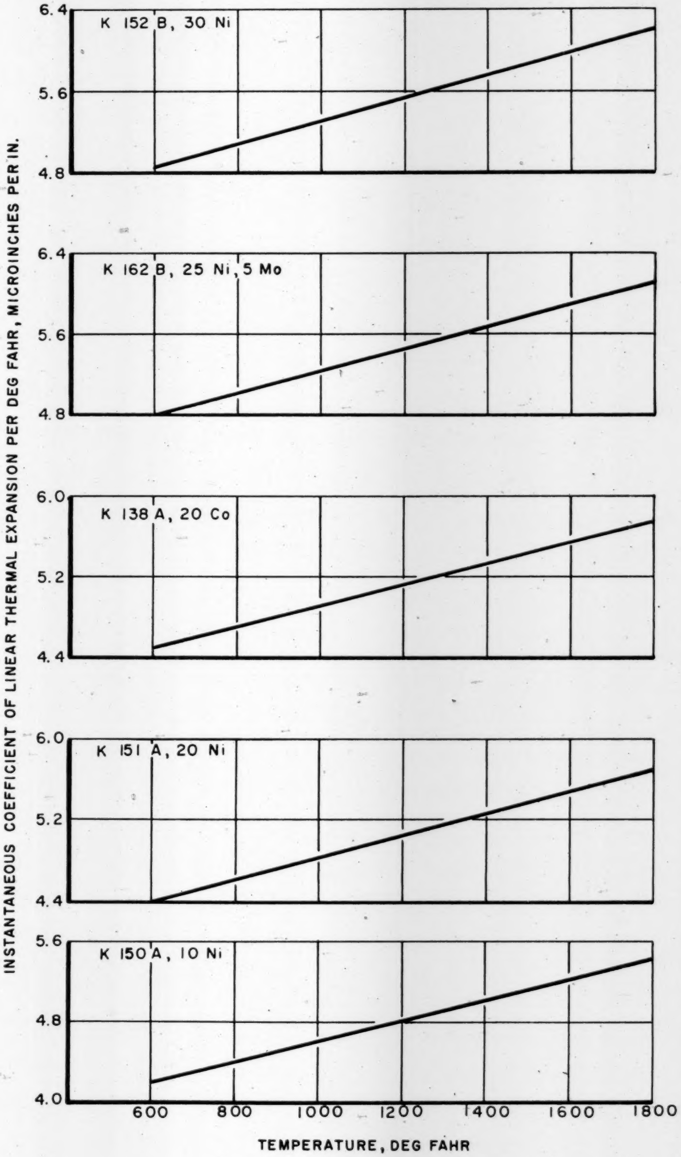

FIG. 6.—Instantaneous Coefficients of Thermal Expansion of Five Titanium Carbide Cermets.

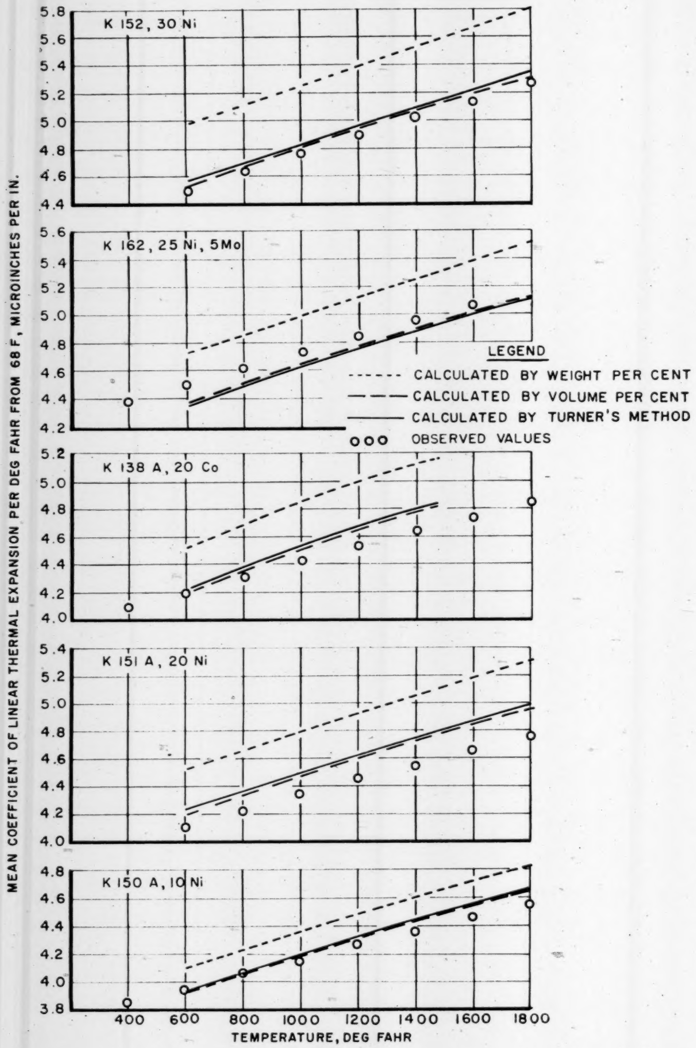

FIG. 7.—Comparison of the Measured Thermal Expansion of Five Titanium Carbide Cermets with That Calculated from Their Compositions by Three Different Methods. (Mean coefficients of thermal expansion 68 to temperature deg Fahr.)

TABLE III.—CONSTANTS FOR THE THERMAL EXPANSION VERSUS TEMPERATURE EQUATION.

NOTE.—Equation is $y = A + BT + CT^2$, where y (strain) is dimensionless and T is deg Fahr

	A	B	C
K 150A	-0.247×10^{-3}	3.62×10^{-6}	0.500×10^{-9}
K 151A	-0.282	3.80	0.527
K 138A	-0.275	3.88	0.520
K 162B	-0.294	4.16	0.547
K 152B	-0.302	4.14	0.608

Values for the thermal expansion of hot-pressed titanium carbide (7) are higher than those reported by Mauer and Bolz, while those reported in reference (8) are lower.

TEST RESULTS

The thermal-expansion curves to 1800 F of these five titanium carbide cermets are shown in Fig. 4. Measurements after test showed no permanent change in length. Their mean coefficients of expansion, together with that of their constituents, are shown in Fig. 3. The mean coefficients of thermal expansion of these cermets are compared with those for a group of metals and alloys in Fig. 5, and instantaneous coefficient of expansion for these cermets are given in Fig. 6. The measured mean coefficients of expansion of these cermets are compared in Fig. 7 with the coefficients calculated from the expansions of the constituents by three different methods. The calculated values were obtained by weighting the expansions of the constituents according to (1) the weight per cent, (2) the volume per cent, and (3) Turner's stress method. The constants for the least squares parabola obtained for the thermal expansion curve of each cermet are given in Table III.

The thermal expansion data determined in this study are compared with data reported by others in Table IV. The compositions of the cermets reported in references (9) and (10) differ slightly from cermets used in this study. The difference between the measured expansions is in the direction to be expected from the difference in composition.

SUMMARY

The thermal expansions of five titanium carbide cermets were measured and an attempt made to find a method for computing their thermal expansions from the expansions of the constituents. The values calculated by weight per cent were from 5 to 11 per cent higher than the observed values. The method presented by Turner (1) and the method based upon volume per cent were found to yield values that agreed with measured values within ±5 per cent. This is considered a good approximation since values reported for various specimens of TiC by one observer (8) differ by ±4 per cent, and values reported by other observers differ more widely. The thermal expansion of hot-pressed TiC bodies (7) showed a thermal expansion from room temperature to 1100 F which was 2 per cent higher than that measured by Mauer and Bolz (6). The thermal expansion of two hot-pressed TiC bodies measured by Engberg and Zehms (8) showed expansions from room temperature to 1000 C which were 9 to 11 per cent below that of Mauer and Bolz. Using an expansion value for TiC 10 per cent lower than that used in our calculations would lower the calculated Turner value approximately 6 to 8 per cent. Using a value for α of TiC 4 per cent lower than Mauer and Bolz would give calculated values for all these materials within ±3 per cent of the observed.

Knudsen, Moreland, and Geller (9,11) reported thermal expansion data for K 151A and K 138A. Their specimens apparently contained 2 per cent less binder and 5 per cent more mixed carbides than those studied in this paper, and therefore had lower expansion. However, the difference in measured expansions was larger than was to be expected from this cause alone. The thermal expansions of K 162B and K 152B reported by Kennametal, Inc. (14–16) are somewhat higher than our measurements. Expansions for 80TiC-20Co measured by Deutsch et al (12) are somewhat higher than measured expansions for K 138A

TABLE IV.—THERMAL EXPANSION DATA FROM OTHER OBSERVERS.

Thermal Expansion, in. per in.

Temperature, deg Fahr	K 151A					K 138A					K 162B		
	Reference (9)				Pratt and Whitney Aircraft	Reference (9)				Pratt and Whitney Aircraft	Temperature, deg Fahr	Reference (15)	Pratt and Whitney Aircraft
	Specimen 1		Specimen 2		Heating	Specimen 1		Specimen 2		Heating			
	Heating	Cooling	Heating	Cooling		Heating	Cooling	Heating	Cooling				
79 to 572	0.0019	0.0013	0.0018	...	0.00206	0.0019	0.0018	0.0018	0.0017	0.00211	RT to 1200	0.00594	0.00549
79 to 752	0.0027	0.0020	0.0027	0.0020	0.00286	0.0027	0.0026	0.0026	0.0025	0.00294	RT to 1600	0.00807	0.00776
79 to 932	0.0035	0.0027	0.0035	0.0029	0.00371	0.0036	0.0035	0.0035	0.0034	0.00379			
79 to 1112	0.0043	0.0035	0.0044	0.0037	0.00459	0.0044	0.0044	0.0043	0.0042	0.00468			
79 to 1292	0.0051	0.0044	0.0053	0.0046	0.00550	0.0053	0.0053	0.0052	0.0051	0.00560			
79 to 1472	0.0059	0.0053	0.0062	0.0055	0.00645	0.0061	0.0062	0.0061	0.0060	0.00656			
79 to 1652	0.0068	0.0061	0.0071	0.0063	0.00743	0.0071	0.0071	0.0070	0.0070	0.00755			
79 to 1832	0.0077	0.0070	0.0080	0.0073	0.00844	0.0081	0.0080	0.0080	0.0080	0.00858			
79 to 2201	0.0093	0.0091	0.0096	0.0093	...	0.0101	0.0100	0.0102	0.0100	...			
Nominal Composition													
Cobalt, per cent	...				...	18				20			
Nickel, per cent	18				20	...				...			
Cb (Ta, Ti) C	15				10	15				10			
TiC	67				70	67				70			

Mean Coefficient of Thermal Expansion per deg Fahr, microinches per in.

Temperature, deg Fahr	K 162B		K 152B	
	Reference (16)	Pratt and Whitney Aircraft	Reference (14)	Pratt and Whitney Aircraft
200 to 1200	5.3	4.94	5.3	4.99
70 to 1200	4.95	4.85	5.0	4.90

Temperature, deg Fahr	Reference (10)		K 138A Pratt and Whitney Aircraft
68 to 1100	4.6	4.9	4.48
Composition:			
Cobalt, per cent	20	30	20
TiC, per cent	80	70	70
Cb (Ta, Ti) C, per cent	...	...	10

20Co-10Cb(TaTi)C-70TiC studied in this paper. A higher expansion is to be expected since the expansion of TiC is greater than Cb(TaTi)C.

The expansions of these five cermets have been compared (Fig. 5) with the expansions of various metals and alloys. This group of cermets could probably be used with metals such as chromium and columbium without difficulties due to differences in expansion.

Acknowledgment:

This work was performed at the CANEL Laboratories, Pratt and Whitney Aircraft, Division of United Aircraft Corp., under contract to the United States Atomic Energy Commission.

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THERMAL EXPANSION OF THIRTEEN TUNGSTEN CARBIDE CERMETS FROM 68 TO 1800 F*

By Lucelia C. Harrington[1] and G. H. Rowe[1]

Synopsis

The linear thermal expansion of 13 tungsten carbide cermets with cobalt binder was investigated experimentally over the temperature range from 68 to 1800 F. Cobalt contents varied from 2.5 to 60 per cent. Several compositions included additions of mixed carbides of titanium, tantalum, and columbium. The experimentally observed coefficients of thermal expansion for the various compositions were compared with coefficients analytically computed from the coefficients for the constituents. Three such analytical methods were evaluated. In one method, the coefficient of expansion of the mixture was computed by volume fractions and in a second method by weight fractions. In the third method, the computation accounted for the stresses set up in the mixture by the difference in thermal expansion of the carbide skeleton and the cobalt binder. The expansions of all these cermets agreed with the values computed by weight fractions or by the stress method within 12 per cent, and by volume fractions within 28 per cent. The cermets containing less than 1 per cent mixed carbides agreed with the expansion computed either by weight fractions or by stress within 8 per cent; those containing more than 5 per cent mixed carbides agreed with values computed by volume fractions within 7 per cent.

The thermal expansions of these cermets are compared with the expansion of some metals and alloys.

Tungsten carbide - cobalt cermets are useful for many high-temperature engineering applications by virtue of their superior strength, hardness, and wear resistance. Typical applications include dies, cutting tools, and bearing materials. For such uses it is desirable to match the coefficient of thermal expansion of a cermet insert with that of the adjoining metal members. The thermal expansions of these cermets were measured to provide information to aid in selecting the proper cermet for a particular design. For tungsten carbide cermets with cobalt binder, the thermal expansion increased with the quantity of binder. The thermal expansion can, therefore, be regulated by the chemical composition.

Total thermal expansion (68 to 1800 F) of these 13 cermets varied from 0.51 to 1.14 per cent. The thermal expansions of such metals as molybdenum, tantalum, columbium, vanadium, chromium, and titanium fall within this range. It is possible to approach the expansion of the nickel-base alloys by increasing the cobalt content of tungsten carbide cermets.

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* Presented at the Sixty-sixth Annual Meeting of the Society.

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TABLE 1.—CHEMICAL COMPOSITION AS GIVEN BY KENNAMETAL, INC.

	Element or Compound, weight per cent						
	Tungsten	Cobalt	Titanium	Columbium	Tantalum	Carbon	Tungsten Carbide
Group 1:							
K11	92.5	2.5	...	...	...	6.0	...
K8	90.3	3.5	...	...	0.3	5.9	...
K10	90	10	...	...	...	...	...
Tungsten carbide - 35 per cent cobalt	...	35	...	...	...	...	65
Tungsten carbide - 60 per cent cobalt	...	60	...	...	...	...	40
Group 2:							
K96	86.3	5.75	...	0.3	1.8	5.8	...
K95	85.6	8.75	...	0.3	0.6	5.6	...
K94	80.6	11.75	...	0.6	1.4	5.4	...
K92	77.0	16	...	0.3	1.6	5.1	...
K91	71.7	20	...	0.9	2.7	4.7	...
K90	65.6	25	...	1.6	3.0	4.4	...
Group 3:							
K3H	65	8.75	11.0	1.8	5.8	7.6	...
K21	65.8	10	6.0	0.6	10.8	6.7	...

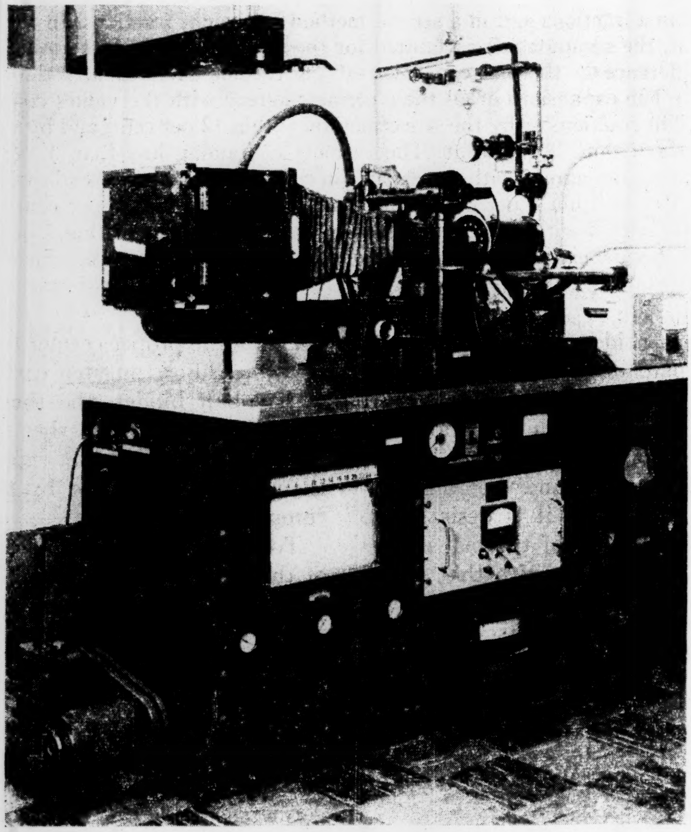

FIG. 1.—Thermal Expansion Apparatus.

Experimental Procedure

Finished machined specimens were purchased from Kennametal, Inc. The nominal chemical compositions in weight per cent in Table 1 are from their advertising literature. Materials tungsten carbide - 35 per cent cobalt and tungsten carbide - 60 per cent cobalt were manufactured at our request. The dilatometer specimens were approximately 0.158 in. (4 mm) in diameter and 1.97 in. (50 mm) long.

Thermal expansion measurements were made with the aid of a Leitz dilatometer (Fig. 1) which uses the autographic optical-lever method with the results recorded on photographic film. This type of equipment is described in ASTM Recommended Practice E 80.[2] The specimen rests in an extended tray of fused quartz. A rod, also of fused quartz, transmits the change in specimen length to a lever that changes the angle of an optical prism. A beam of light reflected from the prism surface traces a line across the photographic film as the specimen expands or contracts. When the furnace is heated, the specimen, the tray in which it rests, and the push rod are all heated and all expand. The push rod and the part of the tray not supporting the specimen are both subjected to the same temperature gradient. Since they are both made of the same material, the errors caused by their thermal expansions will cancel one another. However, a correction must be made for the expansion of the quartz tray under the specimen. This can easily be done since the temperature and thermal expansion of quartz are known.

The specimens were heated in a vacuum (0.06 to 0.3 μ pressure) to prevent contamination. Since heat transfer in a vacuum at temperatures below 800 F was poor, a slower heating rate was used initially followed by a faster heating rate during the remainder of the run. At the beginning of a run, heating was at about 3 F per min and was increased to 5 F per min at the end of heating. With this heating program, it took about 6½ hr to reach 1800 F.

TABLE 2.—DENSITY OF SPECIMENS.

	Density, g per cc		
	Measured	Calculated	Nominal Kennametal
Group I:			
K11	15.20	15.3	15.15
K8	15.10	15.2	15.10
K10	14.38	14.6	...
Tungsten carbide - 35 per cent cobalt	12.22	12.4	...
Tungsten carbide - 60 per cent cobalt	10.67	10.8	...
Group II:			
K96	14.93	14.9	14.80
K95	14.49	14.6	14.60
K94	14.29	14.2	14.10
K92	13.86	13.9	13.81
K91	13.34	13.5	13.41
K90	13.04	12.9	12.90
Group III:			
K3H	10.84	11.1	11.10
K21	12.40	12.4	12.65

The temperature of the specimen was measured by a chromel-alumel thermocouple whose output activated a temperature indicator-recorder. This temperature reading had been found to represent the mean temperature of the specimen within 4 F at 1800 F. Measurements made with three thermocouples welded to a test specimen at center and ends showed that the temperature variation along the specimen length was ±10 F at 1800 F. At every 100 F interval, a break was made in the trace on the film by turning off the light for 1 min. These breaks served as temperature indications on the film. The total expansion was obtained by measuring the height of the expansion curve above the base line at the temperatures at

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[2] Recommended Practice for Dilatometric Analysis of Metallic Materials (E 80), 1961 Book of ASTM Standards, Part 3.

TABLE 3.—CONSTANTS IN EQUATION FOR THERMAL EXPANSION.
$y = A + BT + CT^2$ in which y = thermal expansion, in. per in., and T = temperature, deg F.

	A	B	C
K3H	−0.00022	3.18×10^{-6}	0.50×10^{-9}
K8	−0.00017	2.42	0.32
K10	−0.00023	2.83	0.34
K11	−0.00016	2.33	0.33
K21	−0.00023	3.19	0.39
K90	−0.00024	3.13	0.71
K91	−0.00020	3.09	0.66
K92	−0.00017	2.95	0.49
K94	−0.00019	2.67	0.49
K95	−0.00021	2.79	0.37
K96	−0.00022	2.67	0.29
Tungsten carbide - 35 per cent cobalt	−0.00030	3.99	0.70
Tungsten carbide - 60 per cent cobalt	−0.00035	5.14	0.79

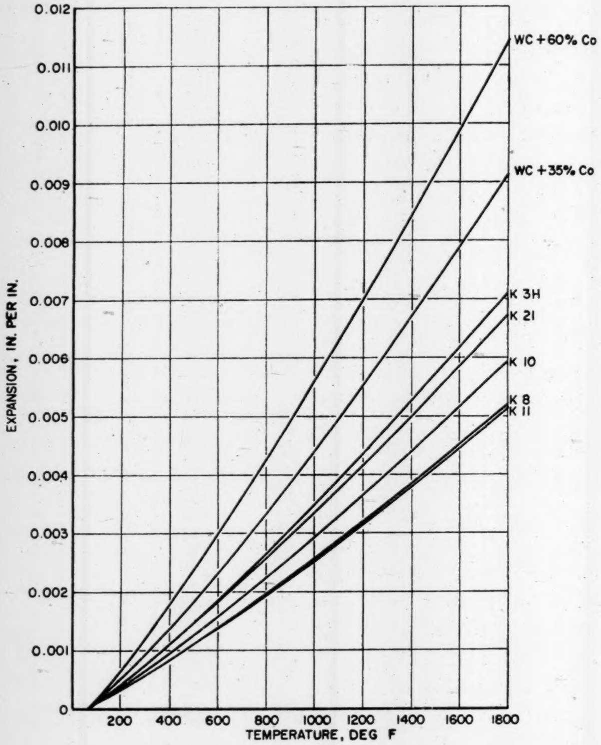

FIG. 2.—Thermal Expansion of Seven Tungsten Carbide Cermets.

which the breaks were made. This gave an expansion value for each 100 F temperature interval. To bring these values into units of inches per inch of specimen length, each was divided by the magnification factor of the dilatometer (approximately 200) and the specimen length. For a specimen 50 mm (1.97 in.) in length, a change of length of 0.001 per cent may be detected. The magnification factor of the instrument is checked about once a year. It has never varied by more than 0.3 per cent over a period of five years. The films on which the expansion is recorded are kept at constant temperature and humidity. A film was chosen which has a minimum dimension change (0.5 per cent or less) during processing and storing.

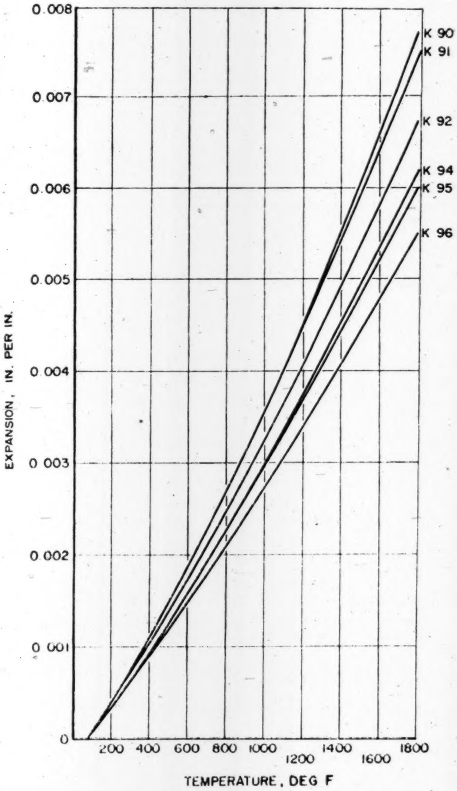

FIG. 3.—Thermal Expansion of Six Tungsten Carbide Cermets.

Before dilatometer measurements were made, the length, diameter, and weight of the specimens were measured. The length and diameter were measured to the nearest 0.0001 in. with micrometer calipers. The specimen was weighed to the nearest 0.0001 g on an analytical balance. The specimen density was calculated from these measurements. Table 2 shows this measured density, the density given by the manufacturer, and the density calculated from the chemical composition.

TEST RESULTS

An equation of the form $y = A + BT + CT^2$, in which y = expansion, in. per in., T = temperature, deg F, and A, B, and C are constants for the material, was fitted to the measured expansions for each material. Table 3 shows values for A, B, and C for each material. At least two runs were made for each material. The above equation was fitted to the data by the method of least squares and fell within the spread of the observed points or was at worst ±2 per cent from the point of poorest fit for all materials at all temperatures above 800 F. The mean coefficient of thermal expansion, α, equals $y/(T\text{-}68)$ and the instantaneous coefficient, dy/dT, equals $B + 2CT$. No significant permanent change in specimen length was observed after test.

The total thermal expansion curves of these 13 tungsten carbide cermets are shown in Figs. 2 and 3. The mean coefficients of expansion of the cobalt and carbide constituents (**1,2**)[3] of these cermets and of the highest and lowest expansion materials of this group are shown in Fig. 4. The range of thermal expansion of these cermets is compared with the expansions of some metals and alloys (**3–6**) in Fig. 5. Figures 6 (*a*) and (*b*) show the mean coefficient of thermal expansion (**1,2,7,8**) versus cobalt content (by volume or weight) for these cermets and for some tungsten carbide - cobalt cermets reported by Gurland and Norton (**7**).

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper.

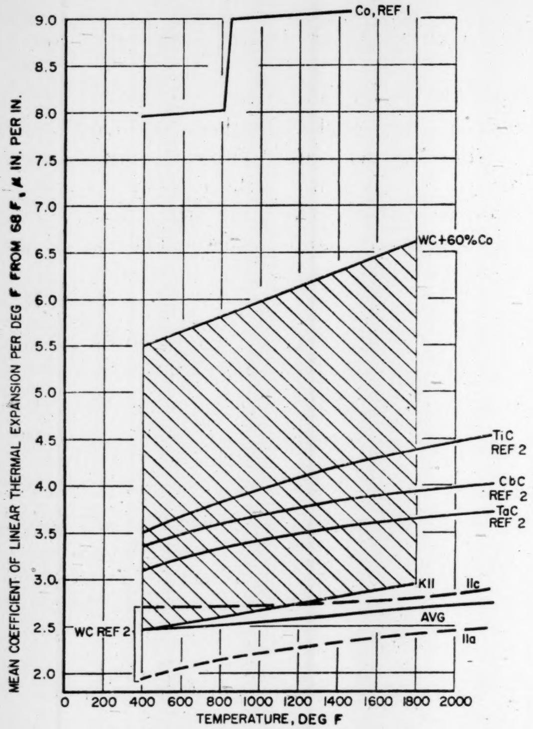


Fig. 4.—Mean Coefficient of Thermal Expansion, from 68 F to t F, for Tungsten Carbide, Columbium Carbide, Tantalum Carbide, Titanium Carbide, and Cobalt Compared with Two Cermets.

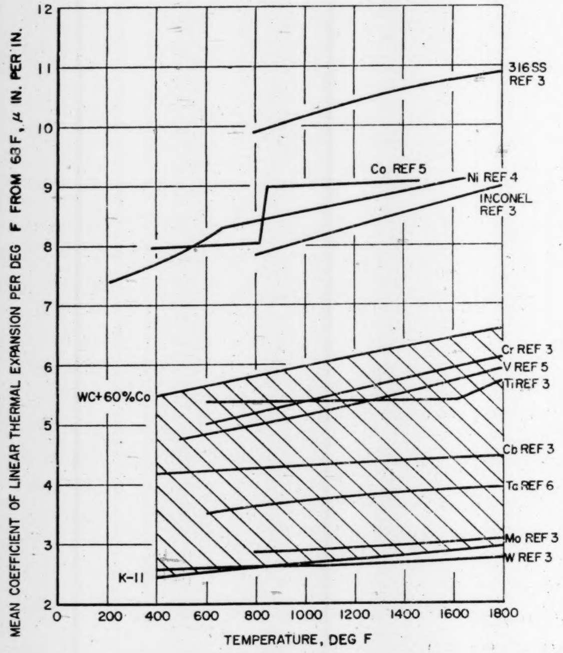
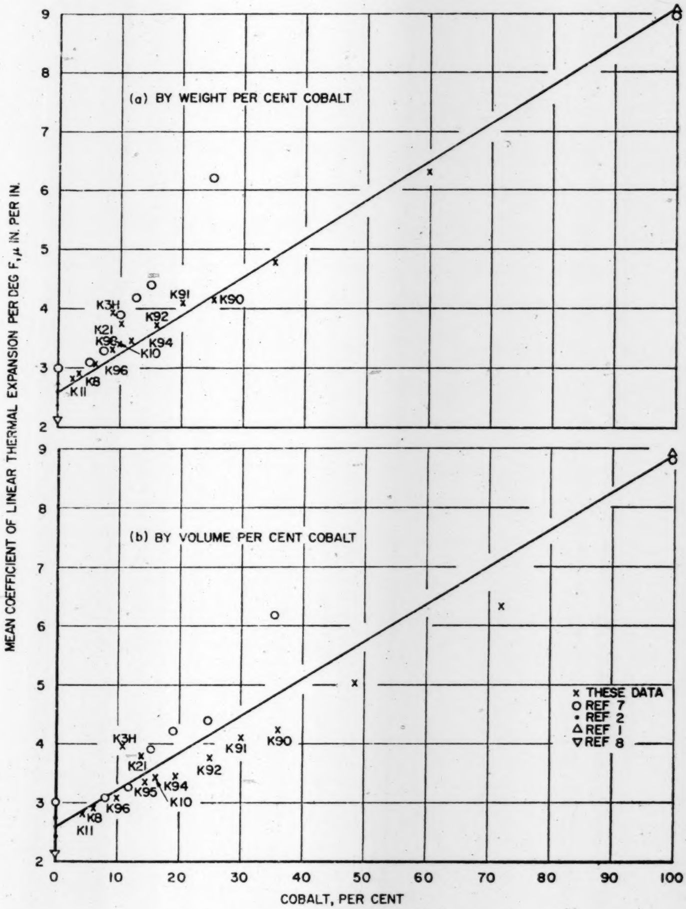


Fig. 5.—Mean Coefficient of Thermal Expansion, from 68 F to t F, of Tungsten Carbide Cermets, Some Alloys and Metals.

Methods by which the thermal expansions of these cermets could be calculated from the expansions of the constituents were evaluated. One method weighted the thermal expansion of each constituent according to its volume fraction in the mixture. A second method weighted the expansions according to the weight fraction of the constituents. A third method used Turner's equation (9) for calculating the average expansion coefficient by taking into account the stresses set up in the mixture by the differences in expansion of the constituents. In Fig. 7 the thermal expansions calculated by these three methods are compared with the measured expansions.



(*a*) By weight per cent cobalt.
(*b*) By volume per cent cobalt.

FIG. 6.—Mean Coefficient of Thermal Expansion, from 68 to 1472 F, Versus Cobalt Content in Tungsten Carbide Cermets.

Table 4 and Fig. 8 compare thermal expansions given in the advertising literature of Kennametal, Inc. (10,11) with our measured values. The difference in the two measurements is seldom more than 2×10^{-4} in. per in.

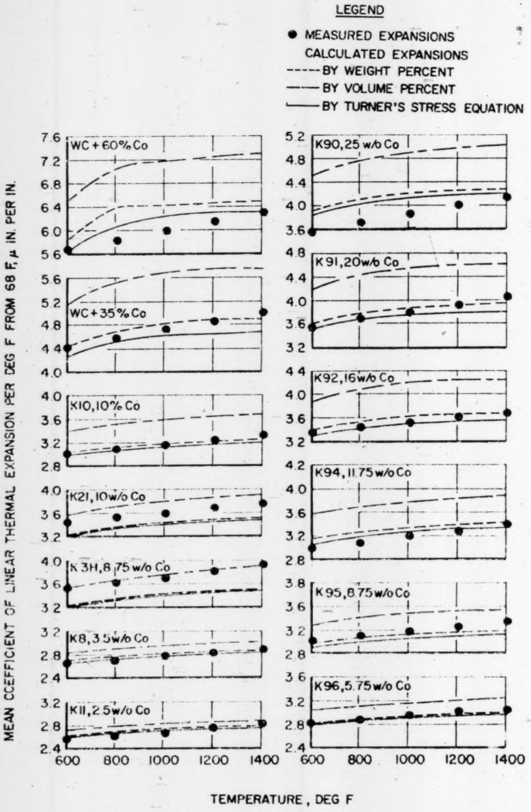


FIG. 7.—Measured and Calculated Coefficients of Thermal Expansion for 13 Tungsten Carbide Cermets.

DISCUSSION

For the cermets containing tungsten carbide, cobalt, and less than 5 per cent mixed carbides, the expansion increased rather linearly with cobalt content (Figs. 6 (*a*) and (*b*)). The two cermets (K3H and K21) that contained from 20 to 22 per cent mixed carbides showed a higher expansion. The thermal expansions of the tungsten carbide - cobalt cermets presented here showed that the composition could be adjusted to approximate the expansion of such metals as tungsten, molybdenum, tantalum, columbium, vanadium, titanium, and chromium. If the tungsten carbide - 60 per cent cobalt cermet were used at 1000 F with Inconel, the difference in expansion would amount to 0.0020 in. per in. For 316 stainless steel, the difference would be 0.0039 in. per in. In some

TABLE 4.—MEAN COEFFICIENT OF THERMAL EXPANSION ROOM TEMPERATURE TO 1200 F OF SOME TUNGSTEN CARBIDE - COBALT CERMETS.
Kennametal - Pratt & Whitney Aircraft Data.

	Kennametal		Pratt & Whitney Aircraft
	Reference (**10**)	Reference (**11**)	
K8	3.0	2.5	2.8
K90	...	3.9	4.0
K92	...	3.4	3.6
K94	...	3.1	3.3
K96	...	2.5	3.0
K3H	3.9	3.9	3.8
K21	3.6	...	3.7

installations these differences might be tolerated.

For evaluating the thermal expansion of these materials, the cermets were divided into three groups: Group 1 (K8, K10, K11, tungsten carbide - 35 per cent cobalt and tungsten carbide - 60 per cent cobalt) contained tungsten carbide, cobalt, and less than 1 per cent mixed carbides (columbium carbide, tantalum carbide); Group 2 (K90 through K96) contained tungsten carbide, cobalt, and from 1 to 5 per cent mixed carbides (columbium carbide, tantalum carbide); Group 3 (K3H and K21) contained tungsten carbide, cobalt, and 20 to 22 per cent mixed carbides (columbium carbide, tantalum carbide, titanium carbide).

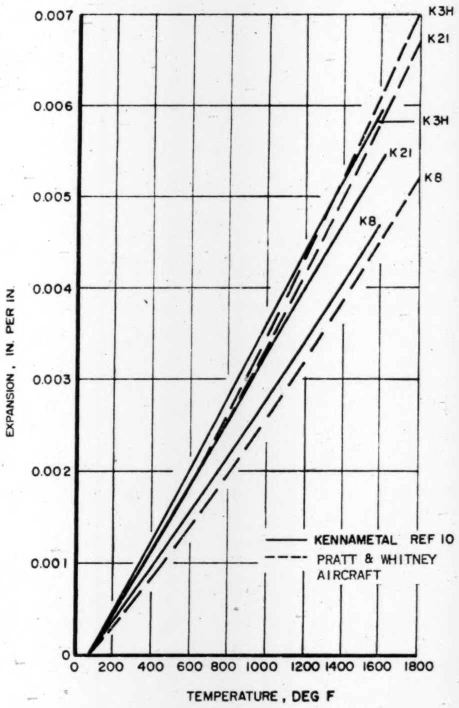


Fig. 8.—Comparison of Linear Thermal Expansion for Three Tungsten Carbide Cermets.

In order to calculate the thermal expansion of mixtures, it was necessary to know the thermal expansion of their constituents. Figure 4 shows the values used in our calculations for the thermal expansion of the constituents. The values for the carbides were from X-ray data of single crystals reported in the literature (2). Expansion values for hot-pressed or sintered tungsten, columbium, tantalum, and titanium carbides (7,8,12–15) are shown in Table 5. For tungsten carbide, the value for mean expansion [$\alpha_{\text{mean}} = (2\,\alpha_a + \alpha_c)/3$] calculated from X-ray data for two crystallographic directions was between those measured by Gurland and Norton (7) and Neels and Pears (8) for sintered or hot-pressed material. For titanium carbide, the expansion measured by X-ray for a single crystal fell between the extremes of values measured on hot-pressed specimens by various observers and methods. For tantalum carbide and columbium carbide, the values measured on the hot-pressed specimens were less than those observed by X-ray for single crystals. Since the quantities of tantalum carbide and columbium carbide in these cermets was small, the value chosen for their expansions had little influence on the computed result. Since the thermal expansion measured by X-ray on single crystals of tungsten carbide and titanium carbide seemed to be representative of all published values, it was decided to use X-ray data (2) for all the carbides. The expansion values used for cobalt were from reference (1). The values chosen for the densities and bulk moduli of these materials are given in Table 6.

Three methods were evaluated for calculating thermal expansions of these cermets—relative volume, weight fraction, and Turner's stress equation. The stress method was that published by Turner (9). His equation was derived by assuming that, at any temperature, the resultant of the forces set up by dis-

TABLE 5.—THERMAL EXPANSIONS OF CERMET CONSTITUENTS.

Material	Specimen Preparation	Method of Measurement	Thermal Expansion, in. per in., at Temperature Interval, deg C								Reference
			Room Temperature to 200	Room Temperature to 400	Room Temperature to 600	Room Temperature to 800	Room Temperature to 1000	Room Temperature to 1200	Room Temperature to 1300	Room Temperature to 1400	
Cobalt	...	...	0.00244	0.00534	0.00928	0.01258	...	...	...	...	(1)
Tungsten carbide	single crystal	X-ray	0.00080	0.00172	0.00267	0.00367	0.00475	0.00584	0.00640	0.00695	(2)[a]
	sintered	dial gage	...	...	...	0.0042	...	...	0.0088	...	(7)
	hot pressed	dial gage	...	...	...	0.0030	0.0042	0.0055	0.0062	0.0069	(8)
Columbium carbide	single crystal	X-ray	0.00109	0.00244	0.00390	0.00538	0.00690	0.00848	...	0.01009	(2)[a]
	hot pressed	dial gage	...	...	0.0028	0.0039	0.0051	0.0064	...	0.0079	(12)
Tantalum carbide	single crystal	X-ray	0.00101	0.00227	0.00361	0.00497	0.00639	0.00786	...	...	(2)[a]
	hot pressed	dial gage	...	...	0.0023	0.0032	0.0041	0.0051	...	0.0062	(12)
Titanium carbide	single crystal	X-ray	0.00113	0.00259	0.00419	0.00594	0.00775	0.00961	...	0.01149	(2)[a]
	hot pressed	direct	...	...	...	...	0.0070	0.0085	...	0.0102	(13)
	hot pressed	direct	...	...	...	...	0.0072	0.0092	...	0.0109	(13)
	hot pressed	interferometer	0.00111	0.00260	0.00427	...	...	...	...	...	(14)
	...	differential transformer	...	...	0.00424	0.00597	0.00791	0.01003	...	0.01241	(15)
	hot pressed	dial gage	...	...	0.0042	0.0057	0.0073	0.0090	...	0.0108	(8)

[a] Mauer and Boltz data corrected from 0 C to 20 C.

similar strains in the constituents on cooling from the sintering temperature must vanish to zero. Turner's method has been previously investigated by Kingery (16) and Merz et al (17). The equation is expressed as follows:

$$\alpha_r = \frac{\dfrac{\alpha_1 P_1 K_1}{d_1} + \dfrac{\alpha_2 P_2 K_2}{d_2} + \cdots + \dfrac{\alpha_n P_n K_n}{d_n}}{\dfrac{P_1 K_1}{d_1} + \dfrac{P_2 K_2}{d_2} + \cdots + \dfrac{P_n K_n}{d_n}}$$

where:

α = mean coefficient of thermal expansion,
P = weight fraction of constituent,
K = bulk modulus at 68 F, and
d = density at 68 F.

Subscripts i (1 to n) and r refer respectively to the ith component and to the resultant mixture. It is interesting to note that Turner's equation reduces to either weight per cent or volume per cent basis depending upon bulk modulus and density values for the constituents. The equation reduces to a percentage by volume if the bulk moduli of the constituents are equal. If the K/d ratios are equal, the result will be proportional to weight fractions. For the cermets in groups 1 and 2 (less than 5 per cent mixed carbides), the K/d ratios were nearly equal or the quantities of the materials with a different K/d ratio (tantalum carbide, columbium carbide) were small. It is, therefore, to be expected that the expansions calculated by weight fraction and Turner's method would be nearly the same. In group 3 (20 to 22 per cent mixed carbides), neither the K's nor K/d ratios were equal. The volume fraction method for calculating the thermal expansions fit the measured data (±7 per cent) better than did the other two methods. For the materials of group 1 (less than 1 per cent mixed carbide), expansions calculated by Turner's method fit the observed data to 7 per cent and expansions calculated by weight fraction fit the observed data by 8 per cent. For the cermets of group 2 (1 to 5 per cent mixed carbides), calculations by Turner's method fit measured data within 9 per cent and calculations by weight fraction fit measured data within 10 per cent.

A search of the literature yielded few thermal expansion values for tungsten carbide cermets. The advertising literature of Kennametal, Inc. gave expansion curves from room temperature to 1600 F for K8, K3H, and K21 (10). It also gave the mean coefficient of expansion from room temperature to 1200 F for K8, K3H, K90, K92, K94, and K96 (11). Figure 8 and Table 4 compare the values for Kennametal, Inc., and show reasonable agreement with our measured values for the above cermets.

TABLE 6.—DENSITY AND BULK MODULI VALUES OF MATERIALS.

	Bulk Modulus, psi	Density, lb per cu in.	Bulk Modulus/Density
Cobalt	25 × 10⁶	0.322	77.6 × 10⁶
Tantalum carbide	23.1	0.529	43.7
Columbium carbide	27.4	0.283	96.8
Tungsten carbide	50	0.565	88.5
Titanium carbide	25	0.177	141.6

Gurland and Norton (7) reported thermal expansion data for tungsten carbide and six tungsten carbide - cobalt cermets. The expansions measured by Gurland and Norton are compared with the measured values for our cermets in Figs. 6(a) and (b). In Fig. 6(a) the mean coefficients of expansion are plotted versus weight per cent of cobalt, while in Fig. 6(b) the expansion is plotted versus volume per cent of cobalt. The expansions of Gurland and Norton's 5.0 and 7.5 per cent cobalt cermets agreed very well with our cermets of groups 1 and 2. The expansions of the 10, 12.5, and 15 per cent cobalt cermets fell in line with our group 3 cermets. The cermet with 25 per cent cobalt showed an expansion much higher than any of ours.

SUMMARY AND CONCLUSIONS

The total thermal expansion of tungsten carbide - cobalt cermets from 68 to 1800 F may be varied from 0.51 to 1.14 per cent by varying the cobalt content from 2.5 per cent to 60 per cent. This range encompasses expansion of metals such as tungsten, molybdenum, tantalum, columbium, titanium, vanadium, and chromium. Evaluation of methods for calculating the thermal expansion of tungsten carbide - cobalt or tungsten carbide - mixed carbide - cobalt cermets yielded the following results:

1. The thermal expansion of tungsten carbide - cobalt cermets with less than 1 per cent mixed carbides agreed with the expansions computed either by weight fraction or Turner's method within 8 per cent and within 22 per cent by volume fraction.
2. The thermal expansion of tungsten carbide - cobalt cermets with 1 to 5 per cent mixed carbides agreed with expansions computed either by weight fraction or Turner's method within 10 per cent and within 28 per cent by volume fraction.
3. The thermal expansion of tungsten carbide - cobalt cermets with 20 to 22 per cent mixed carbides agreed with the expansions computed by the volume fraction within 7 per cent compared to 12 per cent by weight fraction or Turner's method.

Acknowledgment:

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PROPERTIES OF WELDED HIGH-STRENGTH TITANIUM ALLOY SHEET*

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Synopsis

The high strength-to-weight ratios of currently available titanium alloys suggest their use as structural materials for spacecraft components. Six sheet alloys with a 0.2 per cent yield strength of greater than 150,000 psi were welded with nine different filler wire alloys. A three-point combined free- and guided-bend test and a tension test were used to determine the properties of the weld joints. Weld joints made with low-alloy filler wires were more ductile than those made with higher alloy filler wire. Welding after aging resulted in higher ductility and lower strength values than welding prior to aging. On a relative weldability basis the alloys in decreasing order of weldability would be rated: 2.5Al-16V, B120VCA, 6Al-4V, 3Al-6Mo, 4Al-3Mo-1V, and RS140.

As materials for high-performance aerospace vehicles and components and other engineering structures, the currently available high-strength titanium alloys look promising. At the high strength levels where the greatest gains are realized, and with the present state of knowledge in the field of fusion welding and heat treating of these alloys, the characteristically low weld ductility is a great handicap. The weldability of these alloys has been determined by studying the effects of the various welding process variables. This study was concerned with the effects of filler wire composition and the welding heat treatment sequence on the room-temperature bend and tensile properties.

Alloys Investigated

The six commercially available high-strength alloys selected for this work are shown in Table 1. Four of the alloys used as the parent metal in this study—the 2.5Al-16V, B120VCA, 4Al-3Mo-1V, and the 6Al-4V—are included in the Department of Defense Titanium Alloy Sheet Rolling Program and were supplied by the Bureau of Weapons of the Department of the Navy. The other two parent metal alloys studied, the RS140 and the 3Al-6Mo, were included because they have the same strength-level capabilities. All of these alloys were received in the solution-treated condition with a nominal thickness of 0.040 in.

The nine filler wire compositions include the six parent alloy compositions together with the commercially pure Ti75A alloy, the A110AT alloy, and the Ti-3Al alloy. These are also shown in Table 1. Of the filler wires

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* This presents one phase of research carried out at the Jet Propulsion Laboratory, California Institute of Technology, under Contract NAS 7-100, sponsored by the National Aeronautics and Space Administration.

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used, the Ti75A, A110AT, Ti-3Al, and 6Al-4V alloys were obtained as 0.060-in. diameter wire. The other filler wires were made from $\frac{1}{16}$-in. strips sheared from the alloy sheets. All wires were cold-swaged to approximately 0.04-in. diameter and then chemically etched to approximately 0.032-in. diameter. The etching solution was used at room temperature and consisted of 4 per cent hydrofluoric acid and 40 per cent nitric acid in water.

The room-temperature tensile properties of the sheet alloys, heat treated according to the manufacturer's recommendations, are given in Table 2. A 2 by 0.5 in. gage section specimen was used to obtain these results. The strain rate was approximately 1×10^{-3} per min to the yield point, then a crosshead speed of approximately 0.15 in. per min was used to fracture.

TABLE 1.—COMPOSITION OF TITANIUM ALLOYS.

Alloy	Nominal Composition, weight per cent[a]									
	Aluminum	Chromium	Iron	Molybdenum	Tin	Vanadium	Carbon	Hydrogen	Nitrogen	Oxygen[b]
2.5Al-16V	2.49	...	0.22	...	...	15.99	0.03	0.0131	0.014	0.084
B120VCA	4.3	10.4	...	...	...	12.7	0.06	0.0227	0.01	0.044
6Al-4V (sheet)	6.0	...	0.11	...	...	4.1	0.029	0.008	0.008	0.027
3Al-6Mo	3.45	...	0.183	6.13	...	...	0.03	0.0094	0.017	0.038
4Al-3Mo-1V	4.2	...	0.27	3.3	...	1.2	0.04	0.0082	0.018	0.019
RS140	4.90	2.70	1.05	...	...	...	0.031	0.0116	...	0.010
Ti75A	...	...	0.11	...	...	...	0.04	0.0029	0.011	0.009
Ti-3Al	2.9	...	0.03	...	...	...	0.023	0.006	0.007	0.194
6Al-4V (wire)	6.1	...	0.13	...	...	4.0	0.02	0.01	0.017	0.017
A110AT	5.12	...	0.22	...	2.57	...	0.03	...	0.060	0.341

[a] Supplied by producer.
[b] Determined by authors.

TABLE 2.—ROOM-TEMPERATURE TENSILE PROPERTIES OF HEAT-TREATED 0.04-IN. THICK TITANIUM ALLOY SHEET.

Alloy and Nominal Sheet Thickness, in.	Solution[a] and Aging Treatment	Longitudinal			Transverse		
		0.2 per cent Yield Strength, ksi	Ultimate Tensile Strength, ksi	Elongation in 2 in., per cent	0.2 per cent Yield Strength, ksi	Ultimate Tensile Strength, ksi	Elongation in 2 in., per cent
2.5Al-16V (0.046)	1380 F, 30 min; water quench; 975 F, 4 hr	179.0	193.1	5.0	175.5	186.0	4.75
B120VCA (0.041)	1400 F, 30 min; air cool; 900 F, 25 hr	174.3	194.7	6.25	182.0	195.0	4.0
6Al-4V (0.036)	1660 F, 8 min; water quench; 925 F, 8 hr	157.5	169.5	10.5	155.0	168.5	8.25
3Al-6Mo (0.043)	1550 F, 30 min; water quench; 750 F, 2 hr; 1000 F, 24 hr	171.2	186.0	2.0	174.0	184.0	3.0
4Al-3Mo-1V (0.040)	1650 F, 15 min; oil quench; 925 F, 12 hr	157.1	190.5	8.0	157.5	190.5	7.0
RS140 (0.048)	1520 F, 6 min; water quench; 900 F, 6 hr	159.5	197.3	6.6	167.5	208.0	6.0

[a] All sheets were solution-treated by producer.

Since all the sheets were received in the solution-treated condition, the aging treatment shown in Table 2 was the only treatment given prior to testing. This aging was carried out either in a container held at approximately 10 μ of mercury with a mechanical pump, or in a sealed container which was evacuated to approximately 0.1 μ and then filled to a slight positive pressure with argon. For these conditions, the cooling rate from the aging temperature was approximately 20 F per min, somewhat slower than the air cool recommended by the manufacturers.

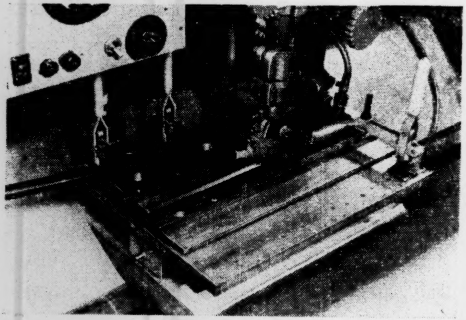

FIG. 1.—Welding Fixture for 0.40-in. Thick Titanium Alloys Sheet.

The 0.2 per cent yield strength for all alloys was greater than 150,000 psi, and for all but the 3Al-6Mo alloy the elongation in 2 in. was 4 per cent or greater.

WELDING PROCEDURE

The automatic inert-gas-shielded tungsten-arc process was used. The filler wire was automatically fed into the arc of a nonconsumable tungsten electrode. The welding process parameters used are given in Table 3, and details of the welding fixture are shown in Fig. 1.

Since the welding torch travel speed and the wire feed are important factors in controlling the dilution of the parent metal during welding, these parameters were carefully controlled to the values given in Table 3. Since all sheets were nominally of the same thickness, a constant welding current could be used without greatly affecting the width of the weld zone, approximately $\frac{3}{16}$ in. on the top surface.

TABLE 3.—PROCEDURES FOR WELDING TITANIUM ALLOY SHEET.

Components	Method and/or Setting
Joint type	Machined-edge butt joint
Joint cleaning	Abrade with steel wool, degrease with acetone
Joint backing	Uncooled grooved copper plate, with 0.030 by 0.375 in. groove
Torch gas flow	30 cu ft per hr of helium
Back-up gas flow	7 cu ft per hr of argon
Trailing-shield gas flow	25 cu ft per hr of argon
Torch travel	14 in. per min
Wire feed	18 in. per min
Wire size	0.032 ± 0.005-in. diameter
Torch and cup	Commercial, water-cooled copper cup 0.75 in. inside diameter on A HW-13 welding torch
Electrode	Pointed 0.040-in. diameter thoriated tungsten, $\frac{1}{4}$-in. extension from torch cup
Arc voltage	20 dc
Arc current	30 amp, straight polarity

To prevent contamination and to ensure a bright weld zone, gas shielding, in addition to that provided by the welding cup and the back-up groove, was provided by a trailing shield $4\frac{1}{2}$ in. long by 2 in. wide with a porous bronze diffusion plate. A chemical analysis of test welds in the 2.5Al-16V alloy gave oxygen contents of 0.08 per cent and nitrogen contents of 0.017 per cent, which are comparable to the interstitials in the as-received sheet (Table 1).

It was therefore assumed that the welding procedure used did not contaminate the weld joint.

TESTING PROCEDURE

Bend Tests:

To provide a preliminary evaluation of the various combinations of alloy and filler wire as well as heat treatment welding sequence, a simple room-temperature bend test was used. This was a combined three-point free- and guided-bend test and used the equipment shown in Fig. 2. It consisted of a 105-deg V wedge with a 0.124-in. radius, which bent the test specimen in a 105-deg V block with a span of 4 in. The bending force was recorded continuously as the output of the load cell. The deflection was determined by a dial gage which measured the relative movement of the testing-machine crossheads.

The test specimen was $4\frac{1}{2}$ by $1\frac{1}{2}$ in. by sheet thickness, with the weld located at the midwidth of the specimen parallel to the $4\frac{1}{2}$-in. dimension and also parallel to the sheet-rolling direction. The specimens were taken from 3 by 14 in. weld panels. Each panel was radiographed to ensure that no defects were present in the test specimen. The weld reinforcement was removed prior to testing, and the top of the weld was on the tension side of the bend specimen.

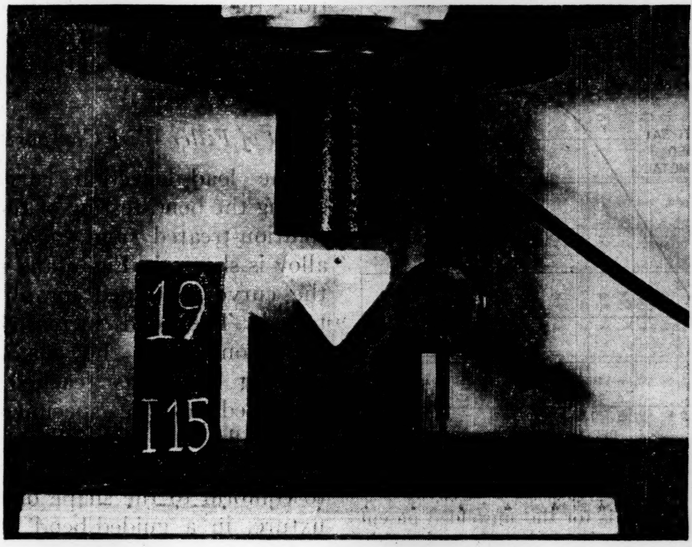

FIG. 2.—Bend-Testing Equipment.

A bend test of this type is particularly well suited for evaluating the ductility of weld joints since it requires each part of the joint to undergo the same deformation and is simpler to make than a tension test with the weld in the longitudinal direction. The combined free- and guided-bend test differs from a free-bend test in that the deflection is continued beyond the point of maximum load for a free-bend test and on into the region of a guided-bend test. In such a test the total deflection prior to fracture is an indication of the ductility of the specimen. Thus, in this bend test the deflection prior to fracture was used to evaluate the ductility of each weld joint, and the maximum free-bend load was used to calculate the modulus of rupture, which is an indication of the strength.

In using the results of this type of test to compare various welds, caution must be used since the fracture deflection values may reflect differences in the strength level, the modulus of elasticity, the thickness, and the residual stress in the specimen.

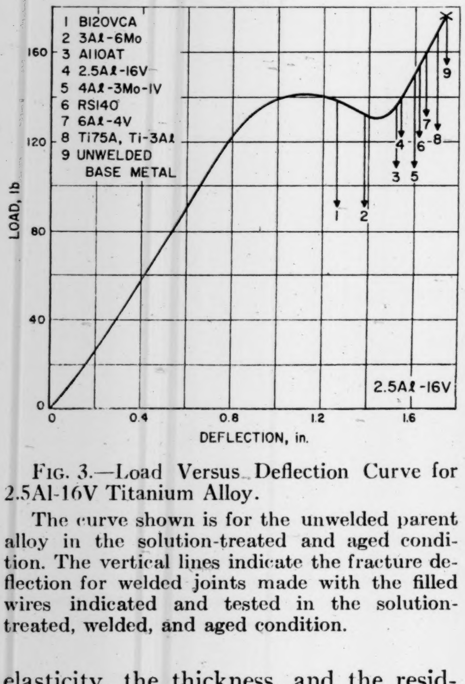

FIG. 3.—Load Versus Deflection Curve for 2.5Al-16V Titanium Alloy.

The curve shown is for the unwelded parent alloy in the solution-treated and aged condition. The vertical lines indicate the fracture deflection for welded joints made with the filled wires indicated and tested in the solution-treated, welded, and aged condition.

Tension Tests:

To evaluate fully the more promising combinations of alloy and filler wire as well as to give design type data, tension tests were run on welded specimens of those combinations that showed the greatest ductility as measured by the bend test. The specimen and testing technique were the same as used for the unwelded tests described earlier.

Tests were run with the weld transverse and longitudinal to the stress direction, and in the solution-treated, welded, and aged condition as well as in the solution-treated, aged, and welded condition. In all cases the weld reinforcement was removed prior to testing, and the stress was applied parallel to the sheet-rolling direction.

TEST RESULTS

For a clearer understanding of the results obtained during this investigation, the effects of the filler wire composition and of the welding heat-treatment sequence will be discussed separately.

Effect of Filler Wire Composition:

The load-deflection curve obtained during the bend-testing of the unwelded, solution-treated and aged 2.5Al-16V alloy is shown in Fig. 3 and is typical of the curves obtained for all the alloys tested. The load increased with the deflection until the maximum free-bending load was reached, and then decreased as the specimen deformed plastically. As the deformation was continued and the specimen was forced to conform to the shape of the bending fixture, in a guided-bend test manner, the load was increased until fracture occurred or until the test fixture bottomed, at which point the test was discontinued.

The general shape of the load-deflection curves for the welded specimens was the same as for the unwelded specimen, except for deflection at fracture. For this reason not all of the curves have been shown. Instead, the fracture deflection for each filler wire for the solution-treated, welded, and aged condition has been indicated on the load-deflection curve for the unwelded, aged parent metal (Fig. 3). The fracture deflection values, as well as the rupture modulus values for all specimens, are given in Table 4.

TABLE 4.—ROOM-TEMPERATURE BEND PROPERTIES OF WELD JOINTS IN HEAT-TREATED TITANIUM ALLOY SHEET.

Filler Wire Nominal Composition	2.5Al-16V Aged at 975 F for 4 hr				B120VCA Aged at 900 F for 25 hr				6Al-4V Aged at 925 F for 8 hr			
	Solution-Treated, Welded, Aged		Solution-Treated, Aged, Welded		Solution-Treated, Welded, Aged		Solution-Treated, Aged, Welded		Solution-Treated, Welded, Aged		Solution-Treated, Aged, Welded	
	Fracture Deflection, in.	Rupture Modulus, ksi	Fracture Deflection, in.	Rupture Modulus, ksi	Fracture Deflection, in.	Rupture Modulus, ksi	Fracture Deflection, in.	Rupture Modulus, ksi	Fracture Deflection, in.	Rupture Modulus, ksi	Fracture Deflection, in.	Rupture Modulus, ksi
Unwelded base metal	1.74	283.9	1.74	283.9	1.70	286.3	1.70	286.3	2.08[b]	235.0	2.08[b]	235.0
Ti75A	1.7	235.7	1.69	217.8	1.46	247.7	1.55	227.9	2.10	234.6	1.84	219.1
Ti-3Al	1.7	...	1.74	218.2	1.54	268.1	1.66	239.4	2.26[b]	230.5	1.82	223.1
6Al-4V	1.65	246.6	1.74	215.7	1.28	270.1	1.70	210.4	...	233.0	1.82	229.9
RS140	1.62[a]	253.6	1.71	215.7	1.30[a]	292.2	1.52	248.5	2.22[b]	257.8	1.78	221.9
4Al-3Mo-1V	1.60	262.3	1.74	209.1	1.54	267.9	1.62	225.0	1.94	249.8	1.68	225.8
2.5Al-16V	1.54	245.2	1.76	213.6	1.02[a]	278.6	1.59	215.4	1.77	233.0	1.62	227.1
A110AT	1.52	283.3	1.74	216.2	1.28	286.5	1.60	230.6	2.24[b]	236.7	1.82	221.2
3Al-6Mo	1.38	262.3	1.70	219.7	1.04	267.1	1.58	235.8	2.10	244.9	1.82	234.7
B120VCA	1.26	257.2	1.78	215.7	1.28	231.2	1.58	230.8	1.38	226.8	1.73	229.7

Filler Wire Nominal Composition	3Al-6Mo Aged at 750 F for 2 hr; 1000 F for 24 hr				4Al-3Mo-1V Aged at 925 F for 12 hr				RS140 Aged at 900 F for 6 hr			
	Solution-Treated, Welded, Aged		Solution-Treated, Aged, Welded		Solution-Treated, Welded, Aged		Solution-Treated, Aged, Welded		Solution-Treated, Welded, Aged		Solution-Treated, Aged, Welded	
	Fracture Deflection, in.	Rupture Modulus, ksi	Fracture Deflection, in.	Rupture Modulus, ksi	Fracture Deflection, in.	Rupture Modulus, ksi	Fracture Deflection, in.	Rupture Modulus, ksi	Fracture Deflection, in.	Rupture Modulus, ksi	Fracture Deflection, in.	Rupture Modulus, ksi
Unwelded base metal	1.58	286.8	1.58	286.8	1.80	278.8	1.80	278.8	1.78	302.9	1.78	302.9
Ti75A	1.16	245.0	1.62	251.5	1.48	254.2	1.53	255.3	1.20	249.4	1.63	260.4
Ti-3Al	0.80	249.2	1.62	243.1	1.46	240.4	1.58	245.3	1.06	...	1.48	267.3
6Al-4V	0.80	231.4	1.54	246.2	1.24	261.0	1.42	254.5	1.15	278.0	1.49	266.2
RS140	0.84	241.4	1.52	251.1	1.28	252.8	1.39	235.1	0.86	273.1	1.34	268.8
4Al-3Mo-1V	0.92	249.5	1.56	255.8	1.36	256.1	1.50	253.6	0.88	289.3	1.48	272.5
2.5Al-16V	0.88	243.1	1.54	247.8	1.32	257.9	1.59	246.6	0.92	290.1	1.55	251.4
A110AT	0.80	220.1	1.18	249.9	1.24	250.7	1.56	246.0	1.03	286.4	1.16	260.8
3Al-6Mo	0.76	232.7	1.56	241.9	1.36	254.0	1.50	237.7	0.92	264.3	1.08	275.4
B120VCA	0.98	263.7	1.60	213.9	0.76	239.8	1.68	231.9	0.69	238.7	1.63	256.5

[a] Value corrected for specimen warpage.

[b] No fracture, test discontinued.

TABLE 5.—ROOM-TEMPERATURE TENSILE PROPERTIES OF WELD JOINTS IN TITANIUM ALLOY SHEET.

All properties measured parallel to rolling direction.
For weld specimens reinforcement has been removed.
Nominal sheet thickness 0.40 in.

Nominal Composition of Alloy	Nominal Composition of Filler Wire	Solution-Treated, Welded, and Aged							Nominal Composition of Filler Wire	Solution-Treated, Aged, and Welded						
		Longitudinal Weld			Transverse Weld					Longitudinal Weld			Transverse Weld			
		0.2 per cent Yield Strength, ksi	Ultimate Tensile Strength, ksi	Elongation in 2 in., per cent	0.2 per cent Yield Strength, ksi	Ultimate Tensile Strength, ksi	Elongation in 2 in., per cent	Location of Fracture[a]		0.2 per cent Yield Strength, ksi	Ultimate Tensile Strength, ksi	Elongation in 2 in., per cent	0.2 per cent Yield Strength, ksi	Ultimate Tensile Strength, ksi	Elongation in 2 in., per cent	Location of Fracture[a]
2.5Al-16V......	unwelded	179.9	193.1	5.0	...	...	...	...	unwelded	179.9	193.1	5.0	...	...	...	...
	Ti75A	157.5	170.5	3.0	183.5	188.9	1.0	H	A110AT	85.9	125.3	6.2	78.1	108.7	3.8	H
	Ti-3Al	85.5	174.5	2.8	180.6	188.3	1.5	H	Ti-3Al	78.5	122.0	6.6	80.9	106.7	3.8	W
	6Al-4V	155.0	172.0	1.0	187.8	196.7	1.5	H	6Al-4V	76.0	120.3	6.0	82.0	108.4	4.0	W
	RS140	156.5	168.7	2.7	180.1	189.1	1.0	H	B120VCA	77.1	118.6	7.0	73.0	106.4	5.0	W
B120VCA......	unwelded	174.3	194.7	6.3	...	...	...	...	unwelded	174.3	194.7	6.3	...	...	...	...
	Ti75A	172.0	182.0	1.5	183.2	201.1	1.8	B	A110AT	121.3	126.2	6.0	123.6	123.6	2.5	W
	Ti-3Al	172.0	175.5	1.2	177.9	192.0	4.3	B	Ti-3Al	119.7	132.3	11.0	128.9	128.9	2.6	W
	RS140	169.3	178.3	2.0	185.6	197.8	2.3	H	6Al-4V	119.5	133.5	12.5	136.8	136.8	2.3	W
	4Al-3Mo-1V	160.7	171.0	1.5	...	...	...	...	4Al-3Mo-1V	118.8	131.9	11.2	127.9	127.9	2.0	W
6Al-4V.........	unwelded	157.5	169.5	10.5	...	...	...	...	unwelded	157.5	169.5	10.5	...	...	...	...
	Ti75A	144.9	156.1	3.8	144.9	147.6	1.0	W	Ti75A	129.3	154.3	4.1	139.6	142.9	1.0	W
	Ti-3Al	144.0	157.0	3.7	149.1	151.2	1.0	H	Ti-3Al	130.2	154.8	4.6	139.2	143.5	1.0	W
	RS140	151.7	161.8	3.0	154.5	161.7	1.5	H	3Al-6Mo	134.6	151.2	2.2	144.5	145.7	1.0	W
	A110AT	148.5	160.5	4.2	154.5	159.8	2.3	W	A110AT	130.7	152.2	4.5	142.3	146.5	1.3	W

3Al-6Mo.	unwelded	171.2	186.0	2.0	. . .	. . .	. . .	. . .	unwelded	171.2	186.0	2.0	. . .	. . .	. . .	. . .
	Ti75A	[b]	164.8	[b]	149.0	149.1	1.3	W	Ti75A	123.0	151.8	2.3	123.2	140.5	1.0	H
	4Al-3Mo-1V	[b]	176.8	[b]	[b]	159.9	[b]	W	4Al-3Mo-1V	120.8	153.6	2.5	125.6	141.6	1.1	W
	2.5Al-16V	[b]	179.8	[b]	170.9	184.2	1.7	W	Ti-3Al	122.7	151.8	1.9	120.1	141.4	1.5	H
	B120VCA	[b]	178.5	[b]	169.8	177.8	2.3	B	B120VCA	101.0	141.7	3.6	114.1	147.6	1.5	H
4Al-3Mo-1V. . . .	unwelded	157.1	190.5	8.0	. . .	. . .	. . .	. . .	unwelded	157.1	190.5	8.0	. . .	. . .	. . .	. . .
	Ti75A	158.0	170.2	1.3	150.3	153.5	1.5	W	A110AT	142.5	165.0	2.0	152.5	160.9	1.5	W
	Ti-3Al	155.8	168.9	1.5	150.1	159.8	1.5	W	Ti-3Al	135.3	162.0	3.2	149.9	158.1	1.3	W
	2.5Al-16V	163.8	176.4	1.7	156.0	182.7	1.8	W	2.5Al-16V	144.7	170.7	2.5	160.0	168.4	1.1	H
	3Al-6Mo	162.4	173.2	1.5	155.3	168.1	1.5	W	B120VCA	146.7	170.9	2.5	156.1	162.8	1.1	H
RS140.	unwelded	159.5	197.3	6.6	. . .	. . .	. . .	. . .	unwelded	159.5	197.3	6.6	. . .	. . .	. . .	. . .
	Ti75A	170.0	186.5	1.0	169.5	199.2	1.5	W	Ti75A	156.8	181.4	2.3	163.2	182.0	1.8	H
	Ti-3Al	166.5	184.0	1.3	172.5	198.9	1.0	W	4Al-3Mo-1V	153.9	177.4	2.3	169.8	185.9	2.0	H
	A110AT	176.7	184.8	1.4	. . .	. . .	. . .	. . .	B120VCA	133.5	156.8	2.2	137.2	140.3	1.0	W
	6Al-4V	179.0	186.0	1.0	170.4	204.0	2.0	W	6Al-4V	155.8	180.8	3.2	166.5	184.1	1.8	H

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[a] B, base metal; H, heat affected zone; W, weld zone.

[b] Fracture occurred before 0.2 per cent yield reached.

The results show that for a given parent metal, this test demonstrates the differences in weld-joint ductility caused by the various filler wires. This is indicated in Fig. 3, which shows results obtained with the 2.5Al-16V alloy. The weld joint made with the B120VCA filler wire fractured shortly after the maximum free-bend load was reached, and therefore exhibited very little ductility. The weld joints made with the Ti75A and the Ti-3Al filler wires did not fracture until a deflection almost equal to the fracture deflection of the unwelded parent metal was reached. Other filler wires produced welded joints with intermediate fracture deflections.

TABLE 6.—RELATIVE WELDABILITY OF HEAT-TREATED TITANIUM ALLOY SHEET.

Nominal Composition of Alloy	Base Metal Ultimate Strength, ksi	Welding, Heat-Treatment Sequence	Relative Weldability[a]	
			Bend Data	Tension Data
2.5Al-16V	193	welded and aged	0.98	0.60
		aged and welded	1.0	1.4
B120VCA	195	welded and aged	0.91	0.32
		aged and welded	1.0	2.0
6Al-4V	170	welded and aged	1.0	0.4
		aged and welded	0.88	0.44
3Al-6Mo	186	welded and aged	0.73	...
		aged and welded	1.0	1.8
4Al-3Mo-1V	191	welded and aged	0.82	0.21
		aged and welded	0.93	0.40
RS140	197	welded and aged	0.67	0.21
		aged and welded	0.92	0.48

[a] Relative weldability $= \frac{\text{Ductility of most ductile joint}}{\text{Ductility of unwelded base metal}}$.

The tension test results for the welded specimens are given in Table 5. The values given are the average of three tests. In the longitudinal weld tests, as in the bend test, each part of the weld area must undergo the same deformation and thus the elongation values are an indication of the ductility of the weld joints. In the transverse weld tests the deformation may occur in the base metal portion of the specimen so that the ductility of the weld joints is not indicated by the elongation values.

The results are in general agreement with those obtained from the bend tests. For all the longitudinal weld tests the ultimate strength and the elongation for the welded and aged condition were lower than for the parent metal. For the aged and welded condition the ultimate strength was lower, but for some combinations the elongation was higher than for the parent metal. For the transverse weld tests the ultimate strength was lower than for the parent metal for all but a few combinations. As indicated (Table 5) for most of the transverse weld tests, fracture occurred in the weld or the heat affected zone and most of the reported deformation occurred in the parent metal.

A study of the bend and tensile data indicated that the composition of the filler wire had some effects common to all parent alloys, in that the larger ductilities were associated with the lower alloy filler wires. The data also permit a rating of the alloys in terms of relative weldability by comparing the ductility of the most ductile weld joint as measured in the bend or the longitudinal tension tests, with the ductility of the parent material. The results obtained by using this method are given in Table 6 where the alloys are listed in order of decreasing weldability and in Fig. 4 where results on the welded and aged materials are presented.

The 2.5Al-16V alloy is listed above the B120VCA alloy because of the higher value obtained from the tensile data in the welded and aged condition. Except for this difference, these two alloys are of nearly equal weldability. The 6Al-4V and the 3Al-6Mo alloys have lower weldability, but there is very little difference between these two alloys. The 6Al-4V alloy is listed first because of the greater elongation values obtained for the longitudinal weld tension tests (Table 5). The relative weldability of the 4Al-3Mo-1V alloy is slightly higher than the RS140 alloy.

It is important to point out that caution should be employed in using this method of comparison based on the bend test data. Such a rating could be affected by the magnitude of the fracture deflection of the parent metal, by the modulus of elasticity of the material, and by the thickness of the test specimen. Since all of these fell into rather narrow limits for the alloys studied, the comparison seems reasonable. In any rating of these alloys consideration must also be given to the strength of the parent metal.

Effects of Welding Heat-Treatment Sequence:

The effects of welding before or after the aging treatment may be determined from the data by comparing the ductility and strength values shown in Tables 4 and 5. For all the combinations studied, except for a few with the 6Al-4V alloy, welding after aging without further heat treatment resulted in greater ductility than welding prior to the aging treatment. This greater ductility probably reflects the presence of a softer, unheat-treated weld zone. This is borne out by the generally lower modulus of rupture (Table 4) and transverse weld ultimate strength (Table 5) values for the solution-treated, aged, and welded specimens. This greater ductility for the aged and welded condition is also shown in Table 6 where relative weldability values equal to or greater than 1.0 are shown for several of the alloys.

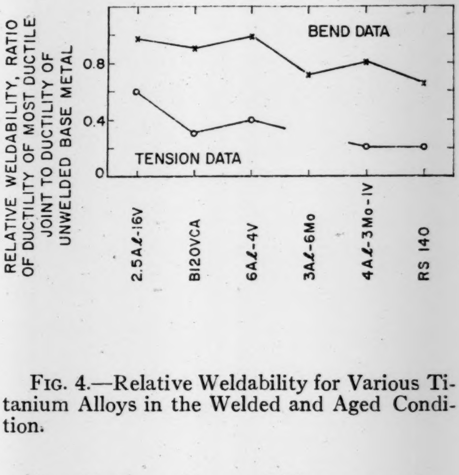

Fig. 4.—Relative Weldability for Various Titanium Alloys in the Welded and Aged Condition.

It is possible that the effects of welding would not be as marked if a different heat treatment was used prior to or after the welding process. A complete metallographic study of each combination of alloy and filler wire would be of help in developing such a heat treatment. It was not within the scope of this work to investigate this area.

CONCLUSIONS

The results of this preliminary study may be summarized as follows:

1. A three-point combined free- and guided-bend test was used to evaluate the ductility of various combinations of base metals and filler wires in weld joints of high-strength heat-treatable titanium alloys. This test used an easy to prepare specimen, and the results were in general agreement with results of longitudinal and transverse weld tension tests.

2. The relative weldability, based on a criterion developed to use the data obtained from this particular bend test, was highest for the 2.5Al-16V and the B120VCA alloys with the former having a slightly higher value. The 6Al-4V and the 3Al-6Mo alloys had lower relative weldability values but were approximately equal to each other. The 4Al-3Mo-1V and the RS140 alloys had still lower relative weldability.

3. For all the parent alloys studied, the weld joints made with the low-alloy filler wires were more ductile at room temperature than those made with the higher alloy filler wires.

4. For all alloys studied, except the 6Al-4V, welding after aging with no further heat treatment gave weld joints of greater ductility but of lower strength than welding prior to the aging treatment.

A DEVICE FOR LOW-COST FATIGUE TESTING*

BY VERNER D. COOMBS[1]

SYNOPSIS

A fatigue testing machine, devised for obtaining data rapidly and at exceptionally low costs, uses a metal ring, rotating about and supported by a cantilever-beam fatigue specimen, to develop a cyclic force on the specimen. The ring is excited to rotate by a motion of the specimen, which is clamped to an oscillating table. This arrangement has permitted simultaneous testing of many specimens at various stress levels. The setting up of the tests has proved to be extremely easy, and inexpensive specimens of many types have been used to obtain data for design purposes.

Optical and strain gage measurements of the imposed cyclic loads have revealed several sources of error. The greatest component of error was introduced by a rotational vibration caused by dimensional inaccuracies in some rings. A mathematical model of the ring motion has been developed as an aid to understanding this behavior. The use of accurately machined rings reduces the error to about 5 per cent.

A fatigue testing machine by which large numbers of fatigue data may be obtained at low cost, with low expenditures of time and effort, was developed at the Locomotive and Car Equipment Laboratory of the General Electric Co., Erie, Pa., to help satisfy demands for more comprehensive information on the fatigue behaviors of engineering materials. These demands include the determination of mean values and confidence limits for fatigue lives and fatigue strengths and investigations on the combined influences of large numbers of factors upon fatigue behaviors of materials.

Approaches to obtaining large numbers of fatigue test data at low cost ordinarily include: (1) the use of many testing machines, or of multistation machines, to reduce the average testing time per specimen; (2) a reduction in the time and effort involved in setting up a test—that is, in positioning and adjusting each specimen in the machine, starting the test, detecting and recording lives, removing failed specimens, calculating stresses, and so on; and (3) a reduction in the cost of preparing each specimen. Multistation fatigue testing machines (machines on which all specimens are cyclically stressed by the same power source) have been used occasionally during the past few decades. Many of these are constant-deflection-amplitude machines employing cantilever beam specimens loaded by crank arm or cam actions (**1–3**).[2]

Setting-up times and efforts are reasonably small for most constant-deflection-amplitude machines, but the requirement of establishing a stress-deflection calibration for each specimen type tested restricts the use of these machines in testing large numbers of specimens that do not conform accurately to a given set of specimen dimensions. This is the case where specimens are parts sampled from production or are sections cut from production parts.

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* Presented at the Sixty-sixth Annual Meeting of the Society.

[1] Metallurgist, Research and Development, General Electric Co., Erie, Pa.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

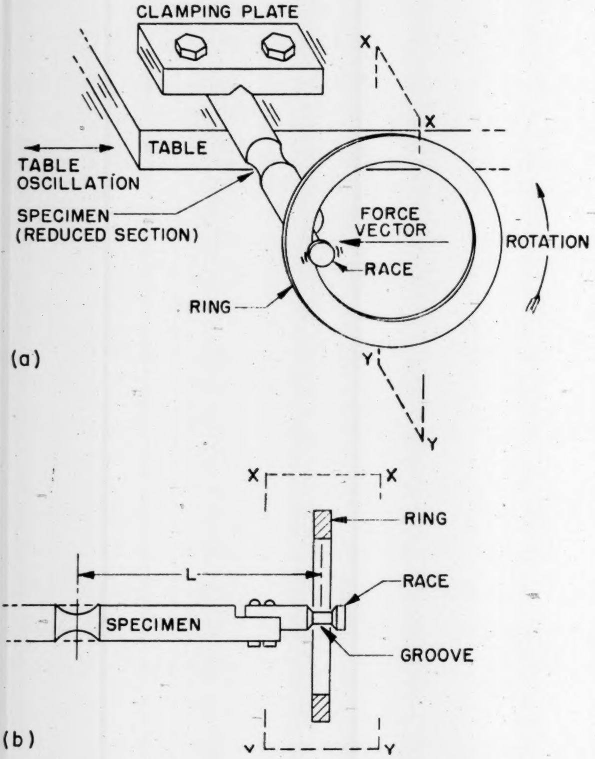

FIG. 1.—Action of the Rotating Ring Tester.

When a constant-load-amplitude machine such as the Sonntag testing machine (4) is used, stress amplitudes can be easily calculated from the machine setting and the appropriate specimen dimensions. However, for each different specimen type some effort is involved in developing means for positioning the specimen for proper coupling to the loading mechanism. In addition, constant-load-amplitude testing is not easily incorporated into a multistation machine design.

The machine described in this paper is the result of an effort to combine more of the advantages of previous machines It is a multistation machine, testing up to 21 specimens simultaneously. The amounts of time and effort required for setting a test are quite small. Inexpensive specimens, often production parts, of widely varying geometries may be tested without modification of machine fixtures.

The novelty of the device is the manner in which each specimen receives the energy required to drive it to fatigue failure. A simple mechanical attachment of the specimen to the machine is used for both gripping the specimen and delivering energy to it. It will be seen that the combinations of advantages mentioned above result from this feature.

DESCRIPTION OF THE DEVICE

The action of the device in fatigue testing a cylindrical cantilever beam specimen is illustrated in Fig. 1(*a*). One end of a specimen is clamped to a part of the machine designated the "table." A metal ring is caused to rotate about the axis of the specimen at the free end, subjecting the specimen to the rotating centrifugal force vector, $\overline{F}$. The force vector produces cyclic reversed bending stresses at all points on the surface of the specimen and ultimately produces failure where these stresses are highest, at the reduced section of the specimen.

As it rotates, the metal ring rolls in the groove of a race attached to the end of the specimen, as illustrated in Figs. 1(*a*) and (*b*). The rotation of the ring is induced by the very slight oscillation of the table, specimen, and race shown in Fig. 1(*a*). The action of the ring is therefore very similar to that of a child's hula hoop. It recovers its frictional energy by extracting energy from the table.

Calculation of the maximum stress amplitude imposed upon the specimen by the device is a simple procedure. The value of the rotating force vector, $\overline{F}$, is:

$$\bar{F} = M\omega^2 R = \frac{4\pi^2 W f^2 R}{g} \quad \ldots\ldots (1)$$

where:

M = the mass of the metal ring, lb-sec² per in.,
ω = the angular velocity of the ring, radians per sec,
R = the radius of rotation of the ring center of mass $\approx$ (Ring inside diameter − Race groove diameter)/2, in.,
W = the weight of the ring, lb,
f = the frequency of table oscillation, cps, and
g = the acceleration of gravity, in. per sec per sec.

The maximum stress amplitude, S_a, exerted on the specimen surface in the region of the reduced section is given by the cantilever beam stress equation:

$$S_a = \frac{FL}{Z} \quad \ldots\ldots (2)$$

where L is the bending moment arm, or distance from the race groove center to the expected crack location, as illustrated in Fig. 1(*b*), and Z is the value of the section modulus at the expected crack location (approximately at the minimum diameter in the reduced section region).

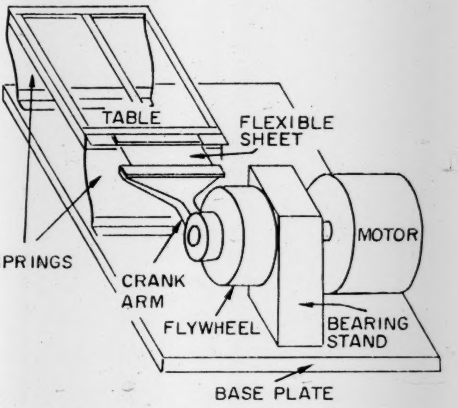

FIG. 2.—Construction of the Testing Machine.

Equations 1 and 2 may be combined to give:

$$S_a = \frac{0.1025\, W f^2 R L}{Z} \quad \ldots\ldots (3)$$

For an established testing machine design (including race and ring design), the values of f and R are fixed. The maximum stress amplitude may be readily calculated from the ring weight, W, and the measured parameters, L and Z, of the particular specimen used. In a like manner, a desired stress amplitude may be obtained by selecting a ring of the appropriate weight. It is noteworthy that the configuration of the specimen, exclusive of the dimensions corresponding to the specimen parameters L and Z, is not important.

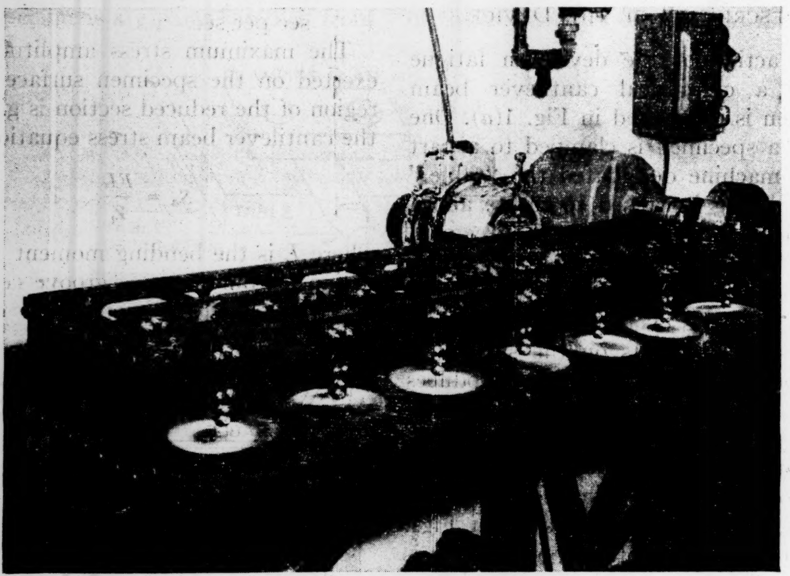

FIG. 3.—Testing Machine in Operation.

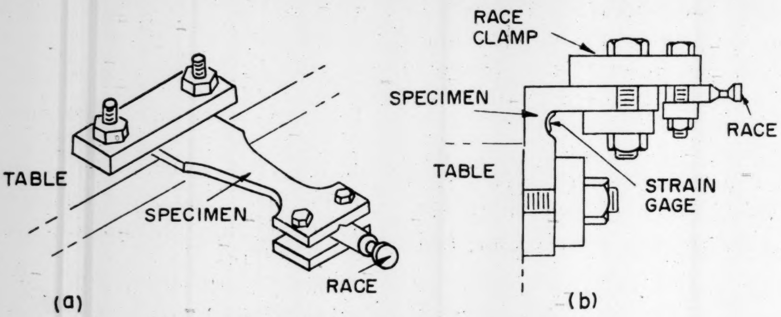

FIG. 4.—Specimens and Mounting Positions.

CONSTRUCTION OF THE MACHINE

A machine constructed to perform the type of testing described in this paper is illustrated in Figs. 2 and 3. It consists of a table, or framework of angle iron, supported above a heavy base of steel plate by means of four sheet springs which allow the table and its specimens one degree of freedom of motion in the horizontal plane. A motor-driven flywheel and crank arm linkage oscillate the table at a displacement amplitude of approximately $\frac{1}{16}$ in. at a frequency of 1800 cpm ($f = 30$ cps). Two belt-coupled motors shown in Fig. 3, a variable speed brush motor, and a synchronous motor, are used alternatively for desirable starting and running speed control. The specimens in the photograph are merely strips of mild steel sheet in which $\frac{5}{8}$-in. radii have been milled to produce reduced section regions. Rings have been set into motion on the front seven of the 21 specimens. The specimens have been mounted in the vertically downward position; however, the only requirement in positioning is that the plane of rotation of a ring contain the direction of oscillation of the table. These specimens have been attached to the table and to the races by means of machine screws. The simplest method of attachment is by clamping, as illustrated in Fig. 4.

Several types of specimens that have been tested are shown in Fig. 4. The types of ring and race illustrated in Fig. 5 are those used in most of the tests.

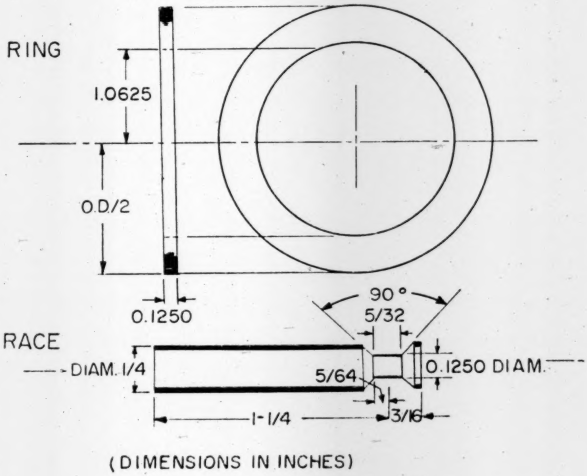
RING
1.0625
O.D/2
0.1250
RACE
DIAM. 1/4
5/64
1-1/4
90°
5/32
0.1250 DIAM.
3/16
(DIMENSIONS IN INCHES)

FIG. 5.—Ring and Race Dimensions.

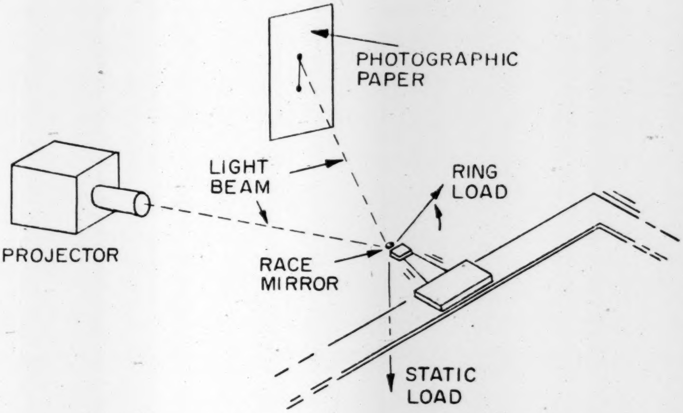
PHOTOGRAPHIC PAPER
LIGHT BEAM
RING LOAD
PROJECTOR
RACE MIRROR
STATIC LOAD

FIG. 6.—Load Measurement by Reflected Light Beam.

OPERATING CHARACTERISTICS

Once the specimens have been positioned, the starting procedure consists of bringing the motor frequency to about

10 rps (table oscillation frequency of 10 cps); a stroboscope has been used for estimating the frequency. The rings are placed into the grooves of the races one by one and are started in spinning by a flick of the fingers, at a rotational frequency greater than 10 cps. As each ring is decelerated by friction to a rotational frequency equal to the table oscillation frequency, the ring falls into synchronism with the table motion, and accepts from the table the energy necessary to offset frictional and windage losses. (An explanation of this phenomenon is included in the Appendix.[3]) When all of the rings are rotating in synchronism with the table motion, the motor speed is increased gradually to the running speed of 1800 rpm. Before the rings are accelerated from their starting frequency a screen is placed around the table so as to contain the rings in the event of a catastrophic failure of a specimen or a race.

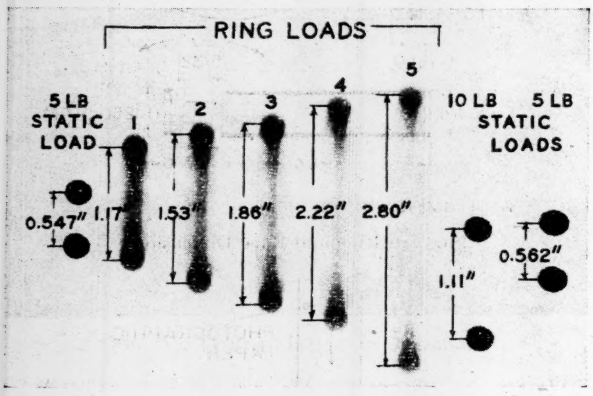

FIG. 7.—Load Amplitude Images from Reflected Light Beam Experiments.

Perhaps the most intriguing feature of the machine is its automatic shutoff characteristic. In tests of ductile materials, the initiation of a crack in a specimen is usually indicated by a falling out of synchronism of the ring rotation with the table motion. Usually the ring slows to a stop and falls from the specimen; occasionally it will coast indefinitely at some fraction of the running speed. This behavior is interpreted to be the result of the drastic increase in power demanded of the ring by the specimen due to the plastic deformation energy absorbed at the root of the crack. If this power exceeds the maximum amount that can be transferred from the table to the ring, the ring cannot maintain synchronism with the table, and thereafter cannot accept energy from it. In the testing of brittle materials, fatigue failure is catastrophic, with the free end of the specimen and the ring being projected in some arbitrary direction (the protective screen provides positive protection for the operator in such testing).

LOAD MEASUREMENTS

Several attempts have been made to determine the agreement of the loads calculated from Eq 1 with those actually imposed upon specimens. Both of these attempts involved comparison of the behaviors of a specimen under a known static condition of load (a dead weight load) and under the calculated cyclic load imposed by a rotating ring.

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[3] See p. 667.

For the first attempt, the equipment illustrated in Fig. 6 was used. A specimen of mild steel similar to that shown in Fig. 4(*a*) was attached to the table in the position illustrated in Fig. 6. To the end of the specimen was affixed a race to which a small circular instrument mirror had been cemented. A collimated beam of light was then projected onto this mirror and reflected to a plane sheet of photographic printing paper. Any vertical deflection of the specimen then caused the reflected beam to sweep the paper vertically, producing a developable line

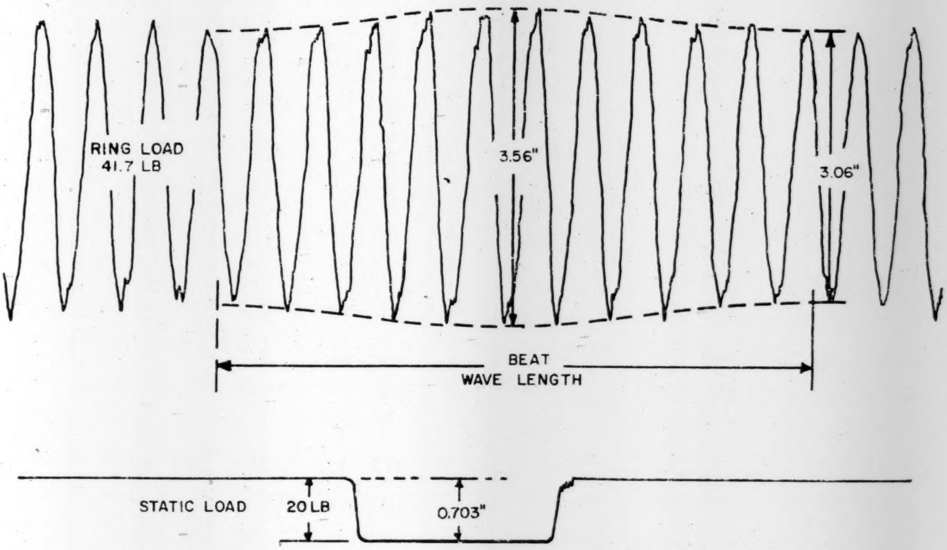

FIG. 8.—Oscillograph Trace Showing Beat in Dynamic Loading.

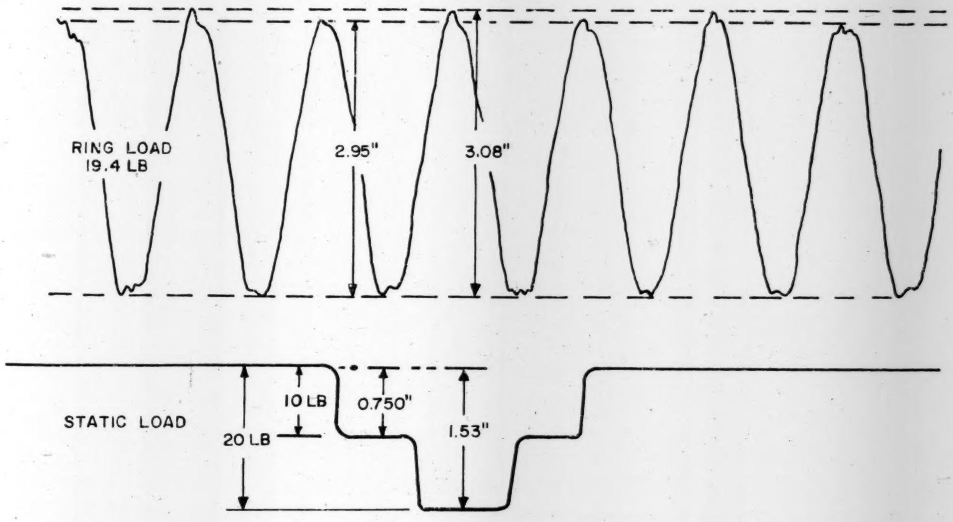

FIG. 9.—Oscillograph Trace Showing Consistent Force-Time Wave.

image of the motion. Several experiments were performed, with the use of known static loads (weights suspended from the race groove) and dynamic (rotating ring) loads to deflect the specimen. The photographic traces for these loads are shown in Fig. 7 and the data from the experiments are reported in Table 1.

The coefficient for relating loads and light beam displacements linearly is available from the static load tests. The value of the actual load amplitude produced by a particular ring is taken to be the same as the value of a static load corresponding to the beam displacement amplitude for that ring. The error in the calculated dynamic loads is seen, in Table 1, to vary approximately from 2 to 7 per cent.

TABLE 1.—LOAD MEASUREMENTS BY OPTICAL REFLECTION.

Ring	Static Loading			Dynamic Loading			
	Weight, lb	Beam Image Deflection, in.	Calibration Coefficient, lb per in.	Calculated Load Amplitude, lb	Beam Image Double Amplitude, in.	Load Amplitude Indicated by Image, lb	Error in Calculated Dynamic Load Amplitude, per cent
No. 1	5	0.547	9.15	...	...	...	...
	...	...	(9.01)	5.65	1.17	5.28	7.1
No. 2	...	...	(9.01)	7.28	1.53	6.90	5.5
No. 3	...	...	(9.01)	8.5	1.86	8.39	1.3
No. 4	...	...	(9.01)	9.98	2.22	10.0	0.2
No. 5	...	...	(9.01)	12.25	2.80	12.6	2.8
	10	1.11	9.00	...	...	...	...
	5	0.562	8.99	...	...	...	...

TABLE 2.—LOAD MEASUREMENTS BY STRAIN GAGED SPECIMENS.

Ring	Static Loading			Dynamic Loading			
	Weight, lb	Beam Image Deflection, in.	Calibration Coefficient, lb per in.	Calculated Load Amplitude, lb	Beam Image Double Amplitude, in.	Load Amplitude Indicated by Image, lb	Error in Calculated Dynamic Load Amplitude, per cent
No. C-3	20	0.703	28.5	...	...	...	...
	...	...	(28.5)	41.7	3.06 to 3.56	43.5 to 51	4.3 to 22
No. R-1	10	0.750	13.3	...	...	...	...
	20	1.53	13.1	...	...	...	...
	...	...	(13.2)	19.4	2.95 to 3.08	19.7 to 20.6	2.1 to 6.5

The second attempt to determine the agreement between actual and calculated dynamic loads employed the strain-gaged specimen of cast iron illustrated in Fig. 4(*b*). As in the previous experiment, the specimen was subjected to both static and dynamic loads. The strain gage outputs for both types of loading were recorded by a direct reading oscillograph. The data obtained in two sets of these tests are given in Table 2, and the oscillograph traces are reproduced in Figs. 8 and 9.

The traces revealed several types of force oscillation superimposed upon the principal force oscillation (that associated with the normal rotation of the ring). One of these additional oscillations is revealed by a beat in the amplitude of the principal force oscillation. This beat, with a frequency of about $2\frac{3}{4}$ cps, is evident in the trace of Fig. 8, but not in the trace of Fig. 9. In addition, the beat could be seen acting on the ring itself by the use of a stroboscope tuned to the tester operating frequency (30 cps). While the ring would be expected to appear motionless under the stroboscope light, it actually exhibits a rotational oscillation, or rocking motion, of about $2\frac{3}{4}$ cps; this motion has been arbitrarily called "ring rocking."

Associated with the presence of ring rocking is a greater range of error in the calculation of dynamic loads, as demonstrated in the data of Table 2. The frequency of ring rocking corresponds to the expected frequency at which the ring would rotate about its own center as the center rotates about the race. It was deduced that the rocking is due to an eccentricity of the inside and outside diameters of the ring; an elaboration of this idea is presented in the Appendix. An accurate remachining of ring C-3, of Table 2, eliminated the rocking in a later test. The data for the second set of static and dynamic load tests reported in Table 2 show an error range for dynamic load calculations of about 2 to 7 per cent.

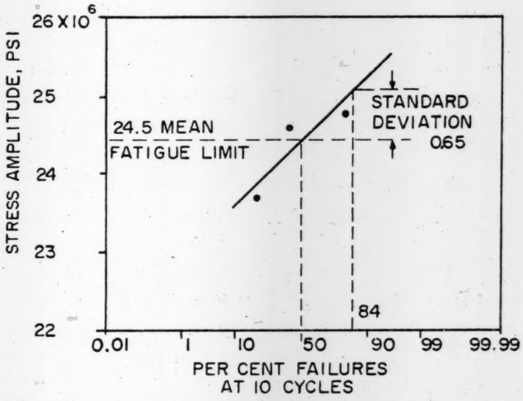

FIG. 10.—Failure Density at Runout Versus Stress Amplitude for Three Groups of Five Specimens Each.

DISCUSSION AND CONCLUSIONS

Applications:

The rotating-ring machine was initially used for the testing of mild steel specimens in a manner somewhat similar to the Probit method (5). Three groups, of five specimens each, were stressed cyclically for 10^7 reversals at three stress amplitudes very near the suspected fatigue limit of the steel. If at least two of the groups exhibited one to four failures after 10^7 cycles, the per cent failure for each group was plotted versus the stress amplitude, on statistical probability paper, and a straight line was fitted to the data. Such a plot is illustrated in Fig. 10. As indicated in that figure, the stress corresponding to the 50 per cent probability intercept of the straight line was used as an approximation of the mean fatigue limit; and the stress difference corresponding to the 84 and 50 per cent intercepts was used as an approximation of the standard deviation of the fatigue limit.

The machine has been employed to a greater extent in rapid comparisons of the finite life fatigue behaviors of nominally similar materials, particularly where the information was required as a basis for a pressing decision on material application. The testing procedure has often been to cut specimens from a production part, drill holes in them to predetermine the fatigue crack locations, calculate the ring sizes for the desired stress amplitudes, attach races to the specimens, clamp the specimens to the table, and start the table and the appropriate rings into motion. In these tests, a rather rapid delivery of fatigue behavior information to engineering sections has been achieved.

The requirements for detecting and recording the lives (numbers of cycles to failure) of individual specimens have varied with the applications. In the tests of mild steel described above, no detection of exact lives was necessary. In the finite life tests, methods of detecting and recording exact lives have depended to some degree on the geometry of the specimens and their modes of ultimate failure—whether ductile or brittle. In the finite life testing of large groups of specimens simultaneously, detection of failures by periodic visual examination is not costly in terms of operator time or of accuracy of the results.

Accuracy:

The agreement of calculated dynamic load amplitudes with actual load amplitudes is estimated from the load measurement tests to be of the order of 5 per cent if the phenomenon known as ring rocking can be avoided. Fortunately this tendency can be detected readily at the initiation of a test by use of a stroboscope and it has been corrected by remachining of rings to correct eccentricities of their inner and outer diameters.

Any factor causing the path of the ring mass center to deviate from the perfect circle assumed by Eq 1 is a source of inaccuracy in the load predicted by that equation. As the table oscillation displacement and the race displacement due to elastic deflection of a specimen usually result in mutually perpendicular distortions of the ring path, attempts were made to match these displacements by appropriate adjustment of the L and Z values of the specimen. The circular path is thereby maintained, but increased slightly in diameter; and the value of R in Eq 1 is increased by the amount of the matched displacement, to serve as a correction factor.

There are other, apparently minor, sources of inaccuracy in the predicted stress wave imposed by the tester; these are illustrated by the trace of Fig. 9. It is suspected that they may be due to lateral fluttering of a ring, which may be excited by windage or by excessive uneven wear in a ring or race. Fortunately the tendency of the ring to flutter can be detected by visual examination of the test in operation.

Conclusions:

The rotating ring tester has demonstrated its capacity for the rapid setting up and testing in fatigue of large numbers of specimens. Specimens of a wide variety of types may be tested simultaneously at many different cyclic stress levels. The load and stress amplitudes employed are calculated by a simple procedure. Load measurement investigations have indicated that accuracy of these calculations may be held to within 5 per cent. Several sources of error in the calculated load amplitudes have been examined; of these, the phenomenon of ring rocking, or rotational vibration of the ring, appears to be the most significant. Success in suppressing ring rocking has been achieved by the use of accurately machined rings.

APPENDIX

RING MOTION

For explanation of the behavior of the rotating-ring tester, the following model has been found to be of value.

System:

A circular system of coordinates, shown in Fig. 11, has been used to describe the motions of the race and ring axes about the system origin, O. The end projections of these axes upon the coordinate plane are indicated by points A (race) and B (ring). For convenience in developing the model, a circular path of the race axis has been assumed; it is described by the constant angular velocity, ω, and the radius vector, $\bar{r}$, of point A from the origin. The motion of the ring axis is described by the angular velocity, γ, and the radius vector, $\bar{T}$, of point B from the origin. Absolute angular displacements of points A and B are assumed not to be important (due to the circular symmetry of the system), and only the angle, α, between the radius vectors $\bar{r}$ and $\bar{T}$ is considered. The displacement of point B with respect to point A is described by the angle, α', and the radius vector, $\bar{S}$, as illustrated in Fig. 11. Vectors $\bar{T}$ and $\bar{S}$ are considered to be large with respect to vector $\bar{r}$, and therefore are assumed to be approximately equal and parallel to one another. Angle α' is then approximately equal to angle α.

Two additional vectors are necessary to the development of the model: the centrifugal force vector, $\bar{F}$, acting parallel and oppositely to vector $\bar{S}$; and the race velocity vector, $\bar{v}$, acting perpendicularly to vector $\bar{r}$. It can be seen from Fig. 12 that $\bar{v}$ and $\bar{F}$ rotate about the race axis with angular velocities, respectively, of ω and approximately γ.

Normal Operation:

The system is considered to be operating normally when the ring motion is in perfect synchronism with the race motion, that is, where

$$\gamma = \omega \quad \text{(4)}$$

For this condition, the ring draws a fairly constant level of power from the tester table, through the race, to offset the frictional and windage losses of energy of the ring. The power, P, that the race delivers to the ring may be expressed:

$$P = (\bar{F} \cdot \bar{v}) - Fv \sin \alpha \quad \text{(5)}$$

It is obvious, from Fig. 11, that the power expended by the ring tends to decelerate the ring and causes a decrease in the angle α. The diminishing of angle α results in an increase in the power transmitted to the ring, according to Eq 5. It is therefore expected that the angle α will assume some equilibrium and negative value, corresponding to the constant level of power demanded by the ring. It is also expected that if the power expenditure of the ring rises sharply (due possibly to an increase in the rate of energy absorbed by the specimen after crack initiation) the maximum level of power, P, that Eq 5 allows may be exceeded. In this case the ring motion would fall out of synchronism with the race motion, and the ring would slow to a stop.

Ring Rocking:

Under conditions where the ring motion does not follow the race motion faithfully, that is, where

$$\gamma = \omega + \frac{d\alpha}{dt} \quad \text{(6)}$$

the behavior of the ring may be described mathematically by equating expressions for the power transmitted to the ring by the race, and for the time rate of change in the kinetic energy of the ring. (In this situation, frictional and windage energy losses of the ring are assumed negligible, and the instantaneous values of power transmitted are those associated with the rotational vibration. The power flows back and forth from race to ring.) The expression for the power, P, is an approximation of Eq 5 for small values of α:

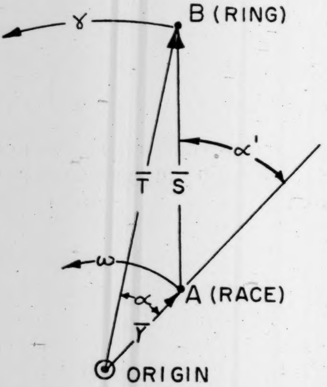

FIG. 11.—Relative Motions of Ring and Race Axes, Demonstrated in Circular Coordinates.

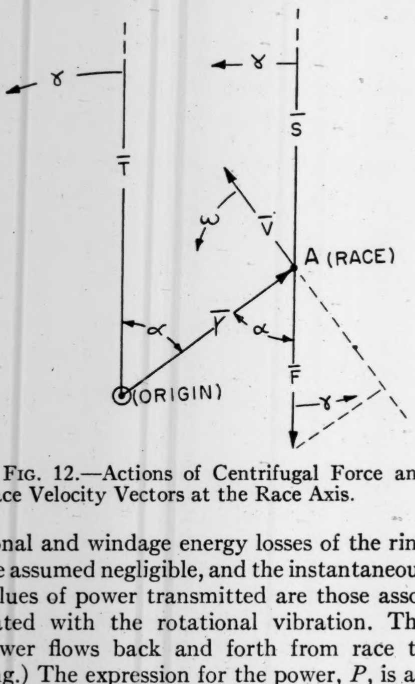

FIG. 12.—Actions of Centrifugal Force and Race Velocity Vectors at the Race Axis.

$$P \approx -Fv\alpha \qquad (7)$$

or,

$$P \approx -F\omega r\alpha \qquad (8)$$

The instantaneous value of the centrifugal force vector is:

$$F = M\gamma^2 R \qquad (9)$$

where M is the mass of the ring and R is the radius of rotation of the ring center of mass about the origin (for a geometrically perfect ring, $R = T$).

Equations 8 and 9 are combined to give:

$$P = -M\gamma^2 R\omega r\alpha \qquad (10)$$

The kinetic energy, E, of the ring may be expressed:

$$E = \frac{I\gamma^2}{2} \qquad (11)$$

where I is the moment of inertia of the ring about the origin, O.

The moment of inertia, I, may be expressed:

$$I = \frac{M}{8}(D_i^2 + D_o^2 + 8R^2) \qquad (12)$$

where D_i and D_o are, respectively, the inside and outside diameters of the ring. Combining Eqs 11 and 12, and taking the time derivative of the resulting expression for E, yields:

$$\frac{dE}{dt} = \frac{M}{8}\left[(D_i^2 + D_o^2 + 8R^2)\gamma\frac{d\gamma}{dt} + 8\gamma^2 R\frac{dR}{dt}\right] \qquad (13)$$

The expressions for power and time rate of change of kinetic energy, Eqs 10 and 13, are equated to yield:

$$-\gamma r\alpha R\omega = \tfrac{1}{8}(D_i^2 + D_o^2 + 8R^2)\frac{d\gamma}{dt} + \gamma R\frac{dR}{dt} \qquad (14)$$

For the situation described by Eq 6, where the rotational vibration known as ring rocking occurs, the center of mass of the ring does not coincide with the ring axis (at the center of the ring's inside diameter) because the inside and outside diameters of the ring are eccentric by some distance, *e*. Therefore, as the ring rotates about its own axis (as the ring axis rotates about the race axis), the actual value of the radius vector, $\overline{R}$, of the mass center of the ring is not equal to that of the radius vector, $\overline{T}$, of the ring axis, but,

$$R = T + e \cos \nu t \qquad (15)$$

where:

ν = the frequency, radians per sec, at which the ring rotates about its own axis,

= (ω) Diameter of the race groove/Inside diameter of the ring.

Before Eqs 14 and 15 can be combined into a useful expression, several approximations, applicable to the tester and testing situation under discussion, must be made:

$$\tfrac{1}{8}(D_i^2 + D_o^2 + 8R^2)\frac{d\gamma}{dt} \gg \gamma R \frac{dR}{dt} \qquad (16)$$

$$\gamma \approx \omega \qquad (17)$$

$$\frac{1}{D_i^2 + D_o^2 + 8R^2} \approx \frac{1}{D_i^2 + D_o^2 + 8T^2} \qquad (18)$$

The approximation of Eq 16 is employed to drop a term from Eq 14, resulting in the following expression:

$$-\gamma r \alpha R \omega = \tfrac{1}{8}(D_i^2 + D_o^2 + 8R^2)\frac{d\gamma}{dt} \qquad (19)$$

With the use of Eqs 15, 17, and 18, Eq 19 is converted to:

$$\frac{d\gamma}{dt} + \left(\frac{8\omega^2 r}{D_i^2 + D_o^2 + 8T^2}\right) \cdot (T + e \cos \nu t)\alpha = 0 \qquad (20)$$

The time derivative of Eq 6 yields:

$$\frac{d\gamma}{dt} = \frac{d^2\alpha}{dt^2} \qquad (21)$$

Finally, Eq 21 is combined with Eq 20 to give:

$$\frac{d^2\alpha}{dt^2} + (\delta + \epsilon \cos \nu t) = 0 \qquad (22)$$

where:

$$\delta = \frac{8\omega^2 r T}{D_i^2 + D_o^2 + 8T^2}$$

$$\epsilon = \frac{8\omega^2 r}{D_i^2 + D_o^2 + 8T^2}$$

This is a Mathieu equation. A solution for the amplitude of the rotational vibration predicted by the equation is beyond the scope of this paper; the reader is referred to Stoker's *Non-Linear Vibrations* (6) for treatments of the Mathieu equation. Several facts can be noted, however, about the system described by the equation. A number of ranges of instability exist, that is, ranges of value for δ and ϵ, for which the vibration amplitude increases without limit. For Eq 22, a stability analysis (6) suggests that decreasing the eccentricity, *e*, and increasing the radius of rotation, *T*, reduces the sizes of the unstable ranges for values of δ and ϵ.

This model has employed a number of approximations and may not be entirely valid for the tester described in this paper. It was generated to illustrate the mechanics of transfer of energy to the rotating ring, and to illustrate a possible explanation for the major fluctuation, ring rocking, in the expected ring motion.

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(5) D. J. Finney, *Probit Analysis*, 2nd Edition, Cambridge University Press, Cambridge, England, 1952.

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DISCUSSION

MR. R. D. BEHR.[1]—Have determinations been made with strain gages to see whether or not the dynamic strain on the side of a round specimen is the same magnitude as that on the top and bottom of the same specimen? I would expect it to be different.

MR. J. A. BENNETT[2] (*presented in written form*).—The author states that the rings must be accurately concentric to produce constant load amplitudes; could he give a quantitative idea of just how close the dimensional tolerances on the rings must be?

It appears to me that there might be a tendency for the rings to rub the sides of the grooves, thus causing wear. Is there any problem with change in weight of the rings due to wear?

Finally, what happens when a brittle specimen fractures? Does it interfere with the other tests?

MR. VERNER D. COOMBS (*author's closure by letter*).—I believe that Mr. Behr's inference is correct. As indicated in the text we expect that, due to the displacement of the table and the deflection of the specimen, the ring mass center follows a path that is not a perfect circle. In addition, we have observed the variations in the angular velocity, ω, that occur due to ring rocking. These variations in the factors employed by Eq 1 of the text cause variations in the centrifugal force vector, F, acting on the rotationally symmetrical specimen of Fig. 1. The stress amplitude at the surface of this specimen is therefore expected to vary somewhat from the top to the side of the specimen. So far we have not done more than to discover that these variations in the force vector exist, to envision the factors that contribute to the variations, and to establish the order of magnitude of the error between calculated and actual values of the load vector in a particular direction (vertical) for the type of material tests we have been conducting.

With regard to Mr. Bennett's question, we have observed that an eccentricity of more than about 0.002 in., for the inner and outer diameters of the rings, would produce some ring rocking. This eccentricity was a consequence of the machining of the rings; we have not as yet observed development of eccentricity due to wear of the rings. Wear has not caused significant loss of mass of the rings; however we do provide lubrication to the races daily. Again, these comments are based on casual observation. Systematic tests on the influence of the magnitude of eccentricity, and on wear rates in the rings, have not been conducted.

As is mentioned in the written text, a specimen fracturing brittlely is projected in some arbitrary direction. It is a simple matter to position a "chicken wire" cylindrical shield about any test of a brittle specimen in order to contain specimen, ring, and race, and to prevent nterference with concurrent tests.

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[2] Metallurgist, National Bureau of Standards, Washington, D. C.

EFFECTS OF PIN-INTERFERENCE AND BOLT TORQUE ON FATIGUE STRENGTH OF LUG JOINTS*

By A. A. Mittenbergs[1] and L. G. Beall, Jr.[1]

Synopsis

A fatigue investigation was conducted on SAE 4340 steel pin-loaded lug joints and on shear joints of the same size. All specimens were subjected to a combined load consisting of a steady tension component and an alternating reversed cantilever-type bending moment. The fatigue strength of pin-loaded lug joints increased with pin interference. However, this improvement was accompanied by an increase in scatter of the fatigue results for the high-pin-interference specimens. The fatigue strength of shear joints increased with bolt torque (or clamping force). An increase of bolt torque beyond a certain required minimum value resulted in relatively small improvement in the load-carrying capacity of shear joints.

The fatigue strength of lug joints is affected by many factors and cannot be readily predicted from available fatigue data obtained on standard unnotched and notched specimens, therefore fatigue testing of actual joints is required. From fatigue investigations (**1–6**)[2] conducted in the past,[3] it is well known that the fatigue strength of lugs can be improved by pin-interference fits or by tight clamping of the lugs. However, most of these investigations have been on simple lug-type specimens under a unidirectional tension - tension fatigue loading. There are no known data in the open literature on the effects of pin-interference or tight clamping on the fatigue strength of complete lug joints, such as are shown in Figs. 1 and 2, subjected to a combined loading.

This exploratory-type investigation was a part of a larger research program (**7**) directed toward the development and substantiation, by means of experimental fatigue studies, of criteria applicable to the design of pin- and bolt-joined connections used in helicopter blade-retention fittings. Hence, the loads applied to the specimens consisted of a steady tension component (designated T in Figs. 1 and 2) corresponding to the centrifugal force in a helicopter blade-retention fitting and a cantilever-type reversed alternating bending moment ($M = Bl$) corresponding to the flapwise or chordwise bending moment. The bending moment was applied in a plane defined by the pin or bolt axis and the longitudinal direction of the specimen. As indicated in the figures, the clevis end was held rigidly and the loads were applied on the free end of the lever.

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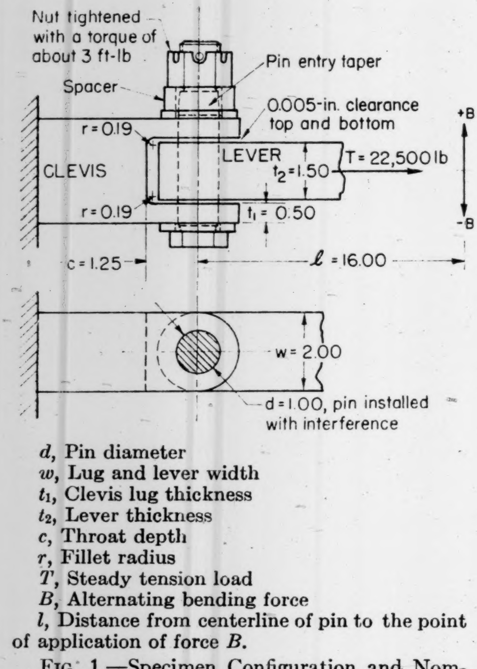
* Presented at the Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] Senior Program Director and Research Engineer, respectively, Solid and Structural Mechanics, Mechanical Engineering Department, Battelle Memorial Institute, Columbus, Ohio.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

[3] Additional references, particularly on some of the earlier investigations, can be found in the references listed in this paper.

It is reasonable to expect that pin-interference fits or tight clamping will improve the fatigue strength of complete joints under the above combined loading, similar to the improvements achieved in simple lugs or complete joints under unidirectional loading. However, these improvements may not be the same quantitatively because of the difference in loading conditions and, therefore, in the interaction of the parts, in stress distributions, and in factors such as pin bending, lug deformations, and fretting. Consequently the purpose of this study was: (1) to investigate the effects of pin-interference fits on fatigue strength of pin-loaded lug joints (Fig. 1); (2) to investigate the effects of bolt clamping forces (or torques) on fatigue strength of shear joints (Fig. 2); and (3) to obtain a comparison between fatigue behaviors of these two types of joints under the same conditions, for the same joint size, and subjected to the combined loading.


Nut tightened with a torque of about 3 ft-lb
Pin entry taper
Spacer
0.005-in. clearance top and bottom
r = 0.19
LEVER
T = 22,500 lb
+B
CLEVIS
t_2 = 1.50
r = 0.19
t_1 = 0.50
-B
c = 1.25
ℓ = 16.00
w = 2.00
d = 1.00, pin installed with interference

d, Pin diameter
w, Lug and lever width
t_1, Clevis lug thickness
t_2, Lever thickness
c, Throat depth
r, Fillet radius
T, Steady tension load
B, Alternating bending force
l, Distance from centerline of pin to the point of application of force *B*.

FIG. 1.—Specimen Configuration and Nominal Dimensions of Pin-Loaded Lug Joints.

For the specimens having low (0.0005 in.) pin-interference, the pins had no entry taper and no spacer was used.

All dimensions are in inches.

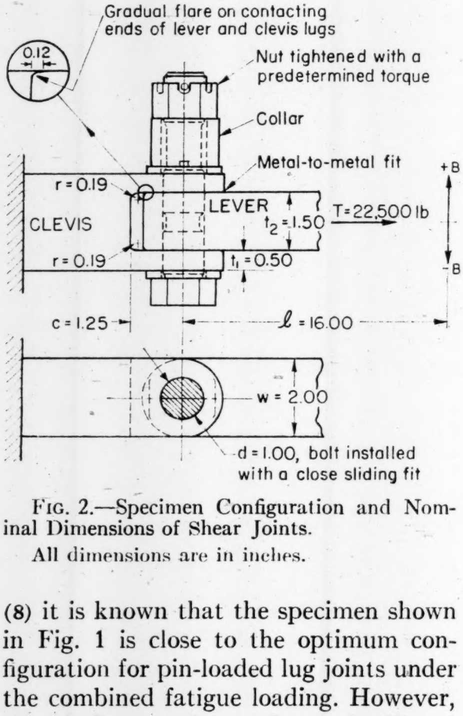

Gradual flare on contacting ends of lever and clevis lugs
0.12
Nut tightened with a predetermined torque
Collar
Metal-to-metal fit
+B
r = 0.19
LEVER
CLEVIS
t_2 = 1.50
T = 22,500 lb
r = 0.19
t_1 = 0.50
-B
c = 1.25
ℓ = 16.00
w = 2.00
d = 1.00, bolt installed with a close sliding fit

FIG. 2.—Specimen Configuration and Nominal Dimensions of Shear Joints.

All dimensions are in inches.

From a previous fatigue investigation (8) it is known that the specimen shown in Fig. 1 is close to the optimum configuration for pin-loaded lug joints under the combined fatigue loading. However, the similar specimen shown in Fig. 2 does not necessarily represent the optimum configuration nor the best design for shear joints. Therefore any comparisons between the results obtained on these specimens (representing the two types of joints) apply to the given conditions only and should not be generalized.

SPECIMENS AND TESTING PROCEDURES

Specimen Designs and Testing Parameters:

All specimens used in this study had the same basic dimensions. The three groups of pin-loaded lug joints tested (Fig. 1) had pin-interferences of 0.0005, 0.002, and 0.003 in., respectively. The pins were installed with the same interference in both the clevis and the lever. Since clearances were provided between lever and clevis lugs, the loads applied on the lever were transferred to the clevis through the pins loaded mainly in bending.

The results presented later in this paper on specimens having 0.0005-in. pin-interference are taken from the previous investigation (8). These specimens (series *B-2*, Fig. 1, and Table I of reference (8)) were the same as those shown in Fig. 1 of this paper except that the pins had no entry taper and no spacer was used. (Figure 2 of reference (8) shows a picture of complete specimens.)

As can be seen from a comparison of Figs. 1 and 2, there are some basic differences in the design details of the two types of joints. In shear joints (Fig. 2) there are no clearances between the lever and the clevis lugs. The bolt is installed with a close sliding fit and its nut is tightened. If the tightening torque is sufficiently high, the applied loads are transferred mainly through the faying (contacting) surfaces of the lever and clevis lugs and not through the bolt. The bolt is preloaded in tension and its primary function is to provide high clamping forces between the contacting surfaces. If for some loading conditions (or low nut torques) the frictional forces between the contacting surfaces are insufficient to transmit the entire load, a slip will occur between the lever and clevis, and a part of the load will then be transferred by the bolt loaded in shear. Under these conditions, considerable surface pressure may develop between the bolt and the bolt hole and some bolt bending may occur.

In the shear-joint specimens, gradual flares were provided across the ends of the contacting surfaces of both levers and clevis lugs to reduce stress concentrations at these locations in the mating element where failures may originate. The bolt had a reduced section inside the lever extending over the middle third of the lever thickness to reduce bolt bending. Another reduced bolt section was added to the bolt length outside the clevis lugs and a collar (see Fig. 2) was placed around the bolt end. Such an arrangement permitted installation of electrical-resistance strain gages on the bolts in the reduced section and determination of axial loads developed in the bolt by the torque applied on the nuts.

To study the effects of clamping force on fatigue strength of shear joints, four groups of specimens were tested, each group having a different torque applied on the bolt nuts. The highest applied torque of 600 ft-lb was believed to be the proper torque for a 1-in. bolt to provide sufficiently high clamping force and, at the same time, not to overload the bolt. The selection of a 300 ft-lb torque for the second group of specimens was based on experimental observation of the behavior of a shear-joint specimen under loading conditions used in this program. This specimen was loaded with the tension load, T, and with the highest alternating bending moment used previously in testing the specimens of the first group (600 ft-lb torque). The clevis lug and lever action was observed with a stroboscope while the bolt torque was gradually increased or decreased. It was seen that above a torque of about 275 ft-lb, the lugs and the lever acted as a unit, whereas below this torque value a separation (breathing) of the parts occurred and a gap appeared between the faying surfaces. Thus the torque of 300 ft-lb, selected for the second group, was slightly above this transition point. The torques of 150 ft-lb and zero for the specimens of the third and fourth groups, respectively, were selected arbitrarily to study the effect of insufficient clamping forces.

Specimen Preparation:

Clevises and levers were fabricated from aircraft quality forged SAE 4340 steel bars of $4\frac{1}{2}$ by $2\frac{1}{2}$-in. cross-section. All material was from the same heat and similarly processed. Blocks of the material were saw cut from the bars and rough machined. They were then quenched and tempered. The average mechanical properties as determined from a number of longitudinal specimens made from heat treated blocks were as follows:

Ultimate tensile strength: 145,000 psi (±5000 psi)
Yield strength, 0.2 per cent offset: 130,000 psi (±4000 psi)
Elongation in 2 in.: 21 per cent (±1 per cent)
Hardness: 32.5 R_C (±1.5 R_C)

The clevises and levers were finished by grinding the outside surfaces of the bars and milling the clevis lugs and lever ends. On shear joints the inside surfaces of the lugs were ground to provide smooth faying surfaces and a close (metal-to-metal) fit between the lugs and the levers. The pin or bolt holes in the clevis and lever were drilled individually in a drill jig with an undersize drill. They were then fitted together in a fixture and line-reamed. All sharp edges were rounded to a radius of about 0.020 in., except that the edges of the holes were deburred and slightly broken (less than 0.005 in.).

Critical dimensions were closely controlled. The largest permissible total variation was 0.002 in. on w, d, and t_1; 0.001 in. on t_2; and 0.010 in. on c. The average surface finish was 5 and 20 rms for ground surfaces and 20 and 100 rms for milled surfaces, parallel and perpendicular to the direction of tool marks, respectively. The average surface finish inside the pin or bolt hole was 80 rms in the direction of the hole axis.

Pins for pin-loaded lug joints were fabricated from SAE 8620 aircraft quality steel. They were carburized to attain finished (after grinding) case depth of about 0.030 in. and heat treated to a case hardness of 58 to 62 R_C. The outside diameters of the pins were ground to give the desired interference fit (0.0005, 0.002, or 0.003 in.) between the pin and the reamed hole for each specimen. The surface roughness on the ground outside pin diameters was below 8 rms.

For the shear-joint specimens, the bolts were fabricated from a modified SAE 4340 alloy steel heat treated to a hardness of 40 R_C (about 180,000 psi ultimate tensile strength). These bolts were ground to such dimensions as to give a close sliding fit in the line-reamed holes (0.0002 to 0.0003-in. clearances) in each specimen.

During assembly of the pin-loaded joints, the clevises and levers were clamped against a straight bar to assure alignment of the assembly. Brass shims were inserted between the lever and the clevis lugs to ensure 0.005-in. clearance on both sides of the lever (these shims were removed after assembly). For specimens with 0.0005-in. pin-interference, the clevis and lever were heated with heat lamps while the pin was cooled in dry ice. A high-temperature antiseize compound containing molybdenum disulfide was applied to the pinhole surfaces prior to heating and one of the two hardened washers (Fig. 1) was placed in position. After installation of the pin, pressure was applied to the pin head by a mechanical press until the joint was frozen. The pins having an entry taper for specimens with 0.002- and 0.003-in. pin-interference were installed with a hydraulic press without heating or cooling the parts. Otherwise the procedures were similar, including application of the same antiseize compound on the pins and pinhole surfaces.

The assembly procedure for shear joints (Fig. 2) was different, since the bolts had a sliding fit. Preassembled specimens with antiseize compound applied on all contacting surfaces were placed in the fatigue-testing machine and a slight tension load was applied on the levers to assure straightness of the specimens. The bolt nuts were then tightened with a torque wrench (the threads were lightly oiled). Prior to assembly, two strain gages (to cancel effects of any bending) were installed on each bolt in diametrically opposite locations on the reduced section under the threads. The instrumented bolts were calibrated in a tension testing machine and a relationship between axial bolt load and strain gage readings was established for each bolt. This relationship was utilized to determine the axial bolt loads (clamping forces) at the various torques applied to the bolt nuts at assembly. The strain-gage leads were carried out through a notch in the collar, as shown in Fig. 3. The relationship between the torques applied and bolt loads developed is shown graphically in Fig. 4. This plot represents the average values obtained. The variations in individual specimens were within 5 per cent of the average.

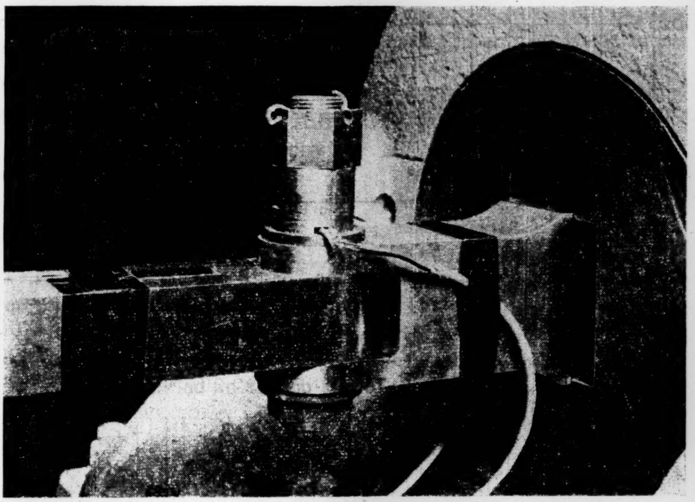


FIG. 3.—Shear Joint Specimen Mounted in the Testing Machine.

Electrical-resistance strain gages installed on top and bottom of the levers were employed for monitoring the applied loads.

The testing machine is described in reference (**8**), which also contains a picture of complete specimens.

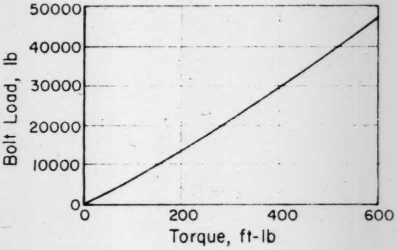


FIG. 4.—Axial Bolt Load as Function of Torque Applied for Shear Joint Specimens (Thread Size: 1 in.—14 NF-3).

Testing Procedures:

The fatigue machines and testing procedures have been described previously (8). In this study, all specimens were subjected to the same steady tension load of $T = 22{,}500$ lb. The alternating (at 1800 cpm) bending moments applied at the centerline of the pin or bolt were varied. The loads imposed on each specimen were controlled and determined by readings from two electrical-resistance strain gages (see Fig. 3) placed on the top and bottom of the lever, 2.5 in. from the centerline of the pin or bolt. For the tension load, each gage was read individually on an SR-4 strain indicator. For the bending moments, the two gages were wired in parallel arms of a bridge to cancel out the steady tension strains and the alternating strains were recorded with a recording instrument.

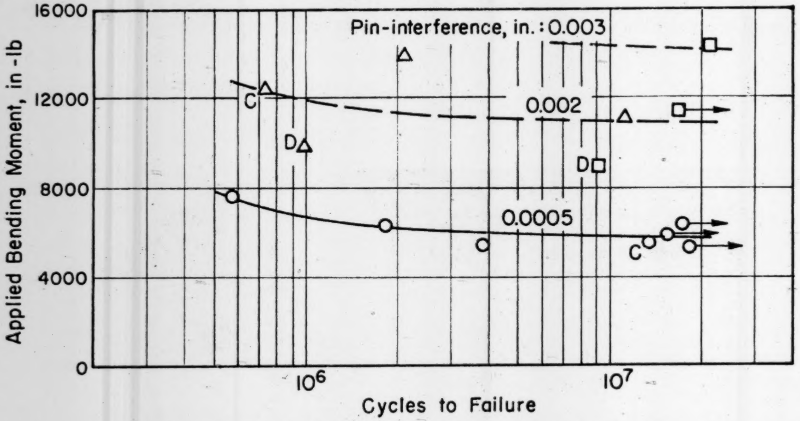


FIG. 5.—Fatigue Test Results for Pin-Loaded Lug Joint Specimens.

All failures occurred in the levers except two clevis lug failures indicated by *C*. Specimens damaged at assembly are indicated by *D*.

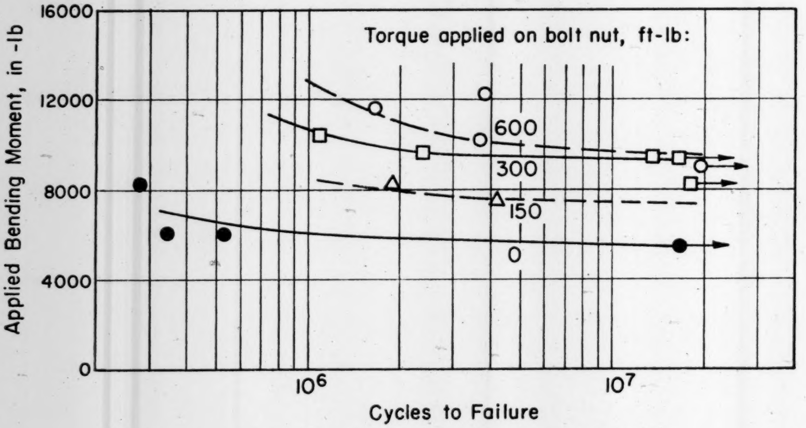


FIG. 6.—Fatigue Test Results for Shear Joint Specimens.

All failures occurred in the levers.

Because of the exploratory nature of this study, only a few specimens (2 to 7) were available in each specimen group. An attempt was made to determine roughly the *M*-*N* (bending moment versus lifetime) curves for each of these groups up to about 15×10^6 cycles. To obtain an indication of the stress distribution in the specimens, strain data were recorded for the outside surfaces of one specimen from each group. (These strain surveys are described in more detail in references (7) and (8).) All tested specimens were disassembled and the fractures and the fretting patterns were examined.

TEST RESULTS AND OBSERVATIONS

Fatigue results of pin-loaded lug-joint and shear-joint specimens are plotted in Figs. 5 and 6, respectively. All data show considerable scatter, as can be expected in testing complete joints, because of the many factors involved that are not present in the usual one-piece specimens.

The exceptionally high scatter for the pin-loaded lug-joints with high (0.002- and 0.003-in.) pin-interference (Fig. 5) is attributable, at least partially, to surface damage occurring in pinholes during assembly of these specimens. On two specimens with high pin-interference, such damage was apparent from metal particles stuck to the pin ends after these were pressed through the clevis lugs and from visible damage (notches) at the lug holes. It is likely that pinhole surface damage was present also in some other high-pin-interference specimens. Disassembly of these specimens usually damaged the pinholes and it was difficult to distinguish whether all damage was done during disassembly or some of it was already present in assembled specimens. The effect of pinhole damage at assembly should be detrimental to the fatigue strength of the joints if the damage occurs at or near the critical peak-stressed locations where failures originate. However, if the damage occurs in some other locations, this may not be the case.

Because of the generally large scatter observed and because of the small number of specimens in each group, some of the M-N curves (shown by dashed lines in Figs. 5 and 6) could not be drawn with certainty from the pertinent test data alone and judgment had to be used in which the results obtained on related specimen groups were taken into consideration. For example, of the three test points shown in Fig. 5 for pin-loaded lug joints with 0.003-in. pin-interference only the highest one appears to be of significance. For these specimens, valid test data at shorter lifetimes could not be obtained because of limitations in testing-machine capacity.

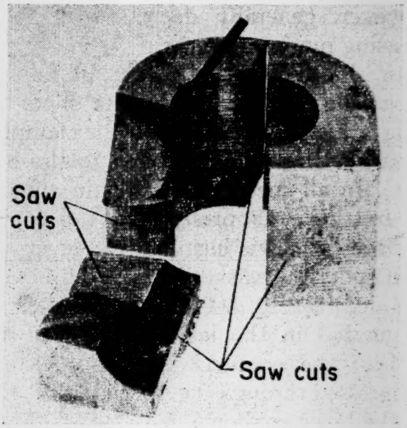


FIG. 7.—Typical Fatigue Fracture of a Pin-Loaded Lug-Joint Lever.

Pin-interference, 0.002 in.; origin of failure indicated by arrow.

During testing of shear joints, strains were recorded in the bolts of several specimens. No significant fluctuations in bolt tension were observed. Also, there was no drop in bolt clamping force apparent after testing.

The mode of failure of pin-loaded lug joints with light pin-interference (0.0005 in.) has been described in some detail in the previous paper (8), which contained also photographs of typical fractures and fretting patterns. The mode of failure of specimens having 0.002- and 0.003-in. pin-interference was generally the same, with all failures originating on the inside of the pinhole. In the majority of pin-loaded lug-joint specimens used in the present investigation, the failure occurred in the levers (two exceptions of failures occurring in the clevis lugs are indicated in Fig. 5). A typical lever failure of a specimen with a 0.002-in. pin-interference is shown in Fig. 7. Fretting was present in the locations of failure origins in all pin-loaded lug-joint specimens. The fretted areas, however, generally decreased with increasing pin-interference.

In shear-joint specimens, all failures occurred in the levers. The mode of failure varied depending on the clamping force (or the torque applied to the bolt nut). In all specimens a certain amount of fretting was present on the faying surfaces of clevis lugs and the lever and also under the washer faces. In specimens torqued up to 600 ft-lb, the failures originated in the lever surfaces at the end of the contacting clevis lugs and progressed through the solid cross-section of the lever as shown in Fig. 8(*a*). These failures were caused by stress concentrations from surface pressure exerted by the flared edges of the clevis lugs on the levers and by the effects of fretting at these locations. There was no evidence of fretting on the bolt or inside the bolt hole.

In specimens torqued up to 300 ft-lb, the failures (Fig. 8(*b*)) still originated in the faying surfaces of the levers and progressed through the solid cross-section of the lever. But the cracks had a tendency to originate closer to the holes and to propagate toward the hole and these cracks intersected the hole surfaces toward the middle of levers. Also, slight traces of fretting were evident on the bolts. Thus these specimens, although still considered representative of sufficiently torqued-up shear joints, showed slight indications of a partial behavior of pin-loaded lug joints.

The mode of failure of the shear joints with 150 ft-lb and zero torques was the same as that of pin-loaded lug joints. The failures originated inside the lever hole and propagated toward the outside. There was somewhat less fretting inside the holes of these specimens in comparison with the pin-loaded lug joints. Part of the alternating load was apparently transmitted through the faying surfaces and these surfaces were fretted.

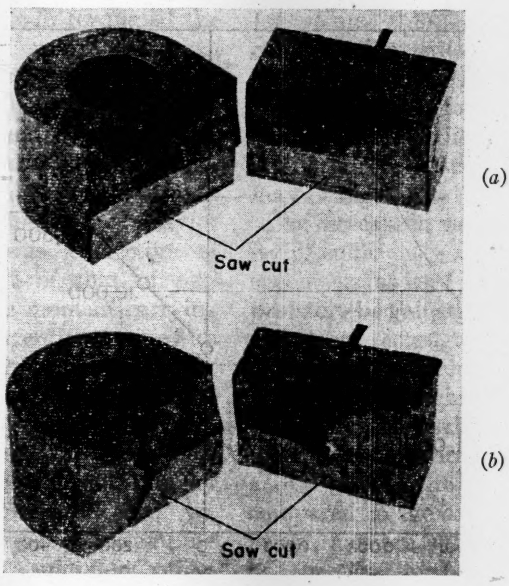
The strain surveys showed that the stress distributions in pin-loaded lug-joint specimens with high pin-interference were more uniform than in those with a pin-interference of 0.0005 in. This indicates that higher pin-interferences decrease pin deflections from applied loads by providing more restraint against pin bending. The indicated stress distributions for sufficiently torqued-up shear joints were, of course, different from those for the pin-loaded lug joints. However, for insufficiently torqued-up shear joints, the stress pattern changed toward that of pin-loaded lug joints.

DISCUSSION OF RESULTS

The effects of pin-interference fits and bolt torques on the fatigue strength of lug joints are shown in Fig. 9 by curves representing the moment-carrying capacity at 15 × 10^6 cycles (taken from Figs. 5 and 6) as functions of pin-interference and bolt torque, respectively. The lifetime of 15 × 10^6 cycles was selected arbitrarily to provide a common basis for comparing and evaluating the test results.

As can be seen from the graph, the moment-carrying capacity of pin-loaded lug joints increased considerably with pin-interference. As compared with the specimens having a pin-interference of 0.0005 in., an interference of 0.002 in. increased the moment-carrying capacity by about 90 per cent and that of 0.003 in. by about 150 per cent. However, the large scatter observed in the test data of the high-pin-interference specimens suggests that caution and conservative approach should be used when employing high-pin-interferences, mainly because of possible damage to pinholes during assembly. Such pinhole damage and an increase in scatter for high-pin-interference connections have been observed also by other investigators (for example, (**1**,**2**,**4**)). Unfortunately the pinhole damage cannot always be readily detected in assembled joints.

Disregarding the possible pinhole damage during assembly, there are several reasons for the marked improvement of fatigue strength in high-pin-interference lug-joints. It can be deduced from the results of a number of photoelastic investigations (**9**–**15**) conducted on lugs and pin-loaded holes under unidirectional loading, with and without pin-interferences, that the main contributing factor toward this improvement is the change (caused by the pin-interferences) in stress distribution within the lugs.



(*a*) 600 ft-lb torque.
(*b*) 300 ft-lb torque.

FIG. 8.—Representative Fatigue Fractures of Sufficiently Torqued-up Shear Joint Specimens. Origins of failures indicated by arrows. Note fretting on the faying surfaces.

In the above photoelastic investigations, the pins were relatively stiff and could be considered as "rigid." For complete pin-loaded lug joints, such as tested in this program, it has been well established (**7**,**8**,**16**) that pin bending from the applied loads contributes significantly to stress concentrations and thus toward lowering the fatigue strength of these joints. As already mentioned, there was less pin bending in the high-pin-interference joints. Therefore, it can be stated that pin-interferences restrain the pin deflections and lower the stress concentrations from pin bending. This also contributes toward the improvement of fatigue strength of pin-loaded lug joints with high-pin-interferences.

A third possible factor contributing toward the improvement of the fatigue strength of these joints is the decrease in fretted areas observed in the high-pin-interference specimens. Such trend may be attributable to the high pressures developed between the pin and pinhole that restrained the pin deflections from the alternating loads. This factor, however, is difficult to evaluate. The fretting at the critical locations where failures originated appeared to be just as severe in the high-pin-interference specimens as in the specimens with 0.0005-in. pin-interference, even if there was a difference in the fretted areas.

The three test data points for the pin-loaded lug joints, lying on a straight line in Fig. 9, are not sufficient to define with certainty the curve characterizing the effects of pin-interference fits in these joints. The shape of the curve shown in the figure is believed to be representative because of the negligible effect of light pin-interferences found by some other investigators (for example, (15)) and because of the results obtained in other investigations on specimens with high pin-interferences. It has been found (for example, (5)) that the fatigue strength of such specimens first increases with the pin-interference, then the increase becomes less pronounced (the curve in Fig. 9 bends to the right), and finally, after the curve reaches its highest point, the ordinates gradually decrease. The latter is the result of overloading the pin holes by high stresses and of possible yielding caused by the very heavy interferences. It is also known (for example, (15)) that there is an optimum value of pin-interference for each specific case under given loading

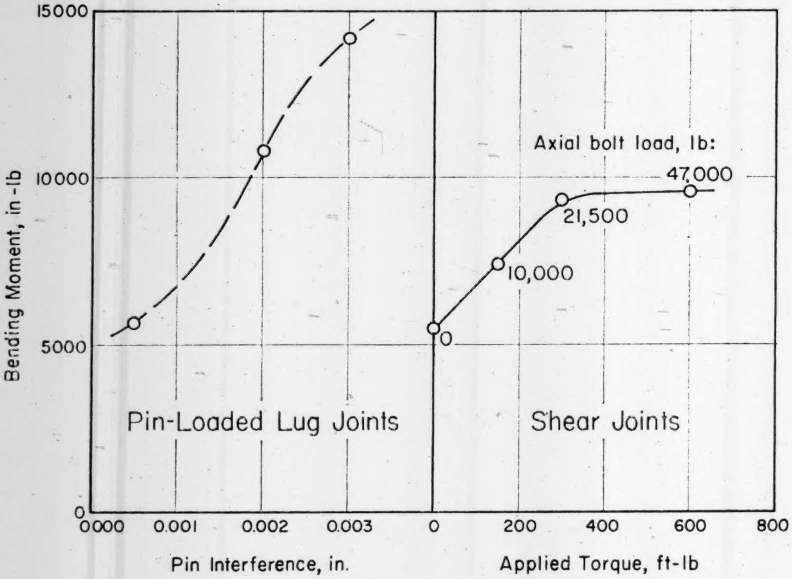


FIG. 9.—Moment-Carrying Capacity of Lug Joints at 15 × 10^6 Cycles.

conditions. For specimens used in this investigation, the optimum interference value is apparently somewhere above 0.003 in.

The moment-carrying capacity of shear joints increased with bolt torque (Fig. 9) as expected. This increase was considerable from a zero torque to a torque value of about 300 ft-lb. A torque value of 600 ft-lb increased the fatigue strength of the shear joints only a little over that at the 300 ft-lb torque. If the experimental observations are also taken into account, it may be stated that the specimens with the 300 and 600 ft-lb bolt torques were representative of true or sufficiently torqued-up shear joints, whereas the zero and the 150 ft-lb torques were insufficient and the fatigue behavior of these specimens was a mixture of true shear joints and of that of pin-loaded lug joints. The moment-carrying capacity of the sufficiently torqued-up shear joints was about 70 per cent higher than that of a shear joint with zero torque.

The results obtained on shear-joint specimens, under the combined loading of this investigation, indicate that an increase in bolt torque (or clamping force) beyond a certain minimum value, required to insure a proper fatigue behavior of a shear joint, results in a relatively small improvement in the load-carrying capacity. Nevertheless it appears to be advisable to overtorque a shear joint if the bolt strength permits, rather than to undertorque it. Higher torques than the minimum value required, in addition to the slight increase in fatigue strength, provide a safety margin against loss of the clamping force under operating conditions (even if no such loss was observed in this investigation).

A comparison of the two curves in Fig. 9 indicates that the moment-carrying capacity of pin-loaded lug joints with high-pin-interferences was greater than that of properly torqued-up shear joints of the same size and that the fatigue strengths of a pin-loaded lug joint with a light pin-interference and of a shear joint with zero clamping force were low and about equal. It should be reiterated, however, that the specimen configuration used in this investigation represented a geometric design that was close to the optimum for the pin-loaded lug joints (7,8). Since this specimen configuration was not the optimum design for the shear joints, it is entirely conceivable that for a different specimen configuration the relative fatigue strengths of these two types of joints may change.

An increase in moment-carrying capacity of sufficiently torqued-up shear-joint specimens, as used in this program, could probably be gained if the lever and clevis ends were rectangular rather than semicircular. The rectangular ends would add some weight, but they would distribute the peak surface pressures at the critical locations over a larger area across the entire specimen width as compared to the area under the gradual flares that are shorter (about one half of the specimen width). There are, of course, some other means of improving the fatigue behavior of shear joints, as well as that of pin-loaded lug joints.

CONCLUSIONS

1. Fatigue strength of complete pin-loaded lug joints under a combined loading increased with pin-interferences, similar to the increase occurring in simple lugs and lug connections under unidirectional loading.

2. Major factors contributing toward the improvement of the fatigue strength in high-pin-interference lug-joints are the changes in stress distribution within the lugs caused by pin-interferences, a decrease in pin bending and the asso-

ciated stress concentrations, and, possibly, a decrease in the detrimental effects of fretting occurring between the pin and pinhole surfaces.

3. Scatter in test results increased with pin-interferences, partially because of damage of pinhole surfaces occurring during assembly.
4. Fatigue strength of shear joints increased with bolt torque (or clamping force).
5. An increase of bolt torque beyond a certain required minimum value resulted in relatively small improvement in the load-carrying capacity of shear joints.

Acknowledgment:

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APPLICATION OF INDUCTION HEATING TO HIGH-TEMPERATURE FATIGUE TESTING*

By D. L. Harper,[1] W. H. Feilbach,[2] and J. F. Libsch[2]

Synopsis

A high-temperature fatigue testing unit which utilizes induction heating has been developed. This unit, permitting simultaneous thermal pulsing and cyclic stressing, consists of: (1) a reversed-bending fatigue testing machine, (2) an induction generator and induction coil, and (3) instrumentation for temperature control. The equipment and its capabilities are described in detail. Fatigue data at 1350, 1500, and 1800 F are presented for type A-286 stainless steel and the data at 1350 F are shown to compare favorably with similar data from literature sources. Fatigue data obtained at 1200 F for type 304 stainless steel also show reasonable agreement with literature available.

Recent activity in the development and application of high-temperature materials has created a demand for new design data. As a result, there is a need for equipment to evaluate the performance of materials under simulated service conditions. Among the properties of interest at elevated temperatures is resistance to repeated loading, that is, high-temperature fatigue. In addition, the influence of thermal pulses or sudden temperature changes in parts cyclically stressed at elevated temperature is important in some applications.

Design of equipment for high-temperature fatigue testing and thermal pulsing involves a number of significant problems. A desirable test system, in addition to being simple and reliable, should provide for independent control of temperature and mechanical stress in the specimen; be capable of testing at extreme temperatures; capable of heating and cooling the specimens rapidly to simulate thermal pulses; and the heating method should have a negligible effect upon crack initiation and propagation. Attempts to use furnace heating and electric-resistance heating of the specimen have failed to attain all of these objectives.

Induction heating can attain high temperatures and is also well suited to simulating rapid thermal cycles. The inherent flexibility of this heating method is due to the fact that the specimen alone is heated. Thus, heating rate is limited only by the available power, and the exposed specimen is cooled rapidly when the power source is deactivated. Maximum cooling rates can be attained with the use of a cooling gas stream. In addition to these advantages, a high-temperature fatigue unit using induction heating can easily be adapted to environmental control.

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* Presented at the Sixty-sixth Annual Meeting of the Society, Atlantic City, N. J., June 23–28, 1963.

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The purpose of this study was to develop and illustrate the capabilities of a high-temperature fatigue testing unit using induction heating and including instrumentation for accurate temperature control.

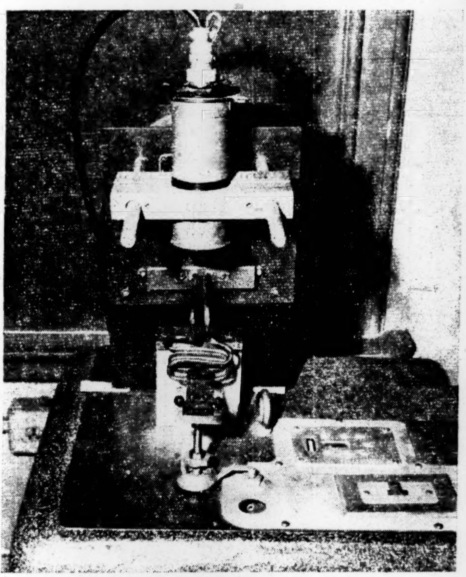

FIG. 1.—Induction Coil of High-Temperature Fatigue Testing Unit.

EQUIPMENT AND PROCEDURES

The high-temperature fatigue unit which has been developed consists of: (1) a reversed-bending fatigue testing unit, (2) a radiation pyrometer, (3) a temperature recorder-controller, (4) a three-function position proportioning control, and (5) an induction generator that energizes an induction coil. The relationship between the induction coil and the specimen is shown in Fig. 1.

A variable reversed-bending load is applied to cantilever specimens by a fatigue-testing machine which can deliver a constant alternating force up to 24 lb at a frequency of 1800 cpm.

A high-frequency induction generator energizes the induction coil which heats the specimen. The induction coil should be close to the specimen for efficient heating, but there must be sufficient room for the specimen to vibrate freely. The four-turn induction coil finally evolved is represented schematically in Fig. 2.

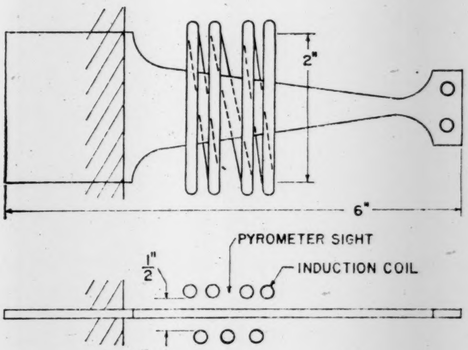

FIG. 2.—Schematic Diagram of Constant-Stress Specimen with Induction Coil.

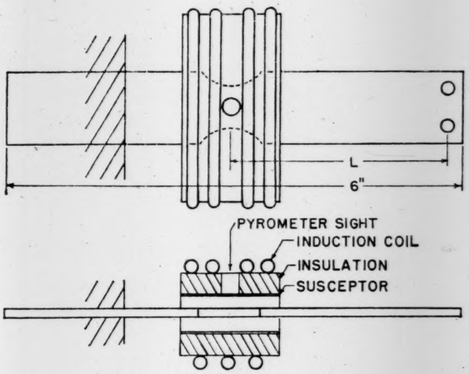

FIG. 3.—Schematic Diagram of Maximum-Stress Specimen with Susceptor-Type Induction Coil.

To avoid direct induction heating of the specimen, a susceptor coil was also designed (Fig. 3). The susceptor, a rectangular Inconel tube with the same interior dimensions as the standard induction coil, is suspended within the induction coil by means of insulating material. In operation, the susceptor is heated by induction while effectively shielding the specimen from the induction field. The specimen is then heated by radiation from the susceptor. This type of heating extends the capabilities of the high-temperature fatigue unit to noninductively heated specimens, such as nonmetallic materials.

The temperature of the specimen is sensed by a radiation pyrometer that sights between the turns of the induction coil onto a target area approximately $\frac{1}{4}$ in. in diameter. The signal from the pyrometer is relayed to a recorder with magnetic modulator positioning control. This unit controls temperature through a mechanical servo-mechanism that can vary the power output of the induction generator.

The proper control setting for each temperature of interest was determined by the use of temperature calibration specimens. These specimens are identical to the test specimens with the addition of thermocouple wires individually welded near the target area by capacitor discharge. When a calibration specimen was installed, the control setting was varied until a potentiometer indicated that the specimen was being held at the desired temperature. This control setting was then used to control the same temperature on standard test specimens. Calibration specimens also were used frequently to verify the control during the testing program. Temperature calibration was ordinarily performed with no specimen vibration; however, tests made on vibrating specimens showed that vibration had a negligible effect with proper sighting of the pyrometer. This calibration technique circumvented the problem of changing emissivity during oxide formation because the final

TABLE I.—CHEMICAL COMPOSITION OF MATERIALS USED IN FATIGUE-TESTING UNIT.

Element, per cent	Material				
	Type 304[a]	Type 304[b]	A-286[c]	A-286[d,e]	Tinidur[e,f]
Carbon	0.061	0.067	0.08	0.045	0.10
Manganese	0.87	0.50	1.25	1.60	...
Silicon	0.60	0.65	0.86	0.65	...
Phosphorus	0.021	...	0.016	0.015	...
Sulfur	0.011	...	0.009	0.010	...
Chromium	18.76	18.21	14.85	15.00	15.00
Nickel	9.53	9.56	24.92	25.25	30.00
Molybdenum	0.19	...	1.26	1.25	...
Copper	0.31	...	...	...	...
Nitrogen	...	0.056	...	...	...
Titanium	...	...	2.03	2.10	1.80
Vanadium	...	...	0.24	0.30	...
Aluminum	...	...	0.28	0.20	...
Boron	...	...	0.007	0.003	...

[a] Source of data, Lehigh University.
[b] Source of data, Cross, see footnote 3.
[c] Source of data, Lehigh University.
[d] Source of data, Roberts et al, see footnote 4.
[e] Nominal composition.
[f] Source of data, Allegheny Ludlum Steel Corp., see footnote 5.

calibration was made on an essentially stable oxide coating. Alternately, graphite suspensions are available to maintain constant emissivity where necessary. However, in the latter instance, there must be certainty that no surface reaction exists between the specimen and the coating.

Tests indicate that there is a $\frac{1}{4}$-in. width across the specimen that is reasonably uniform in temperature and in which failure always occurs. Thermocouples placed around the specimen in this region have shown that there is a maximum variation of ±15 F from the average temperature. In addition, there is a variation of less than ±5 F from the average temperature at any point in the uniform region for the duration of the test.

MATERIALS AND SPECIMEN PREPARATION

The composition of the type 304 stainless steel used in the investigation is given in Table I. Also listed is the composition of the material used for the comparison data reported by Cross.[3] The material was received in the cold-rolled and annealed condition. The specimens were machined and surface ground directly; however, a $\frac{1}{2}$-hr stress-relieving treatment at 1200 F was employed before testing.

The composition of the A-286 alloy, a precipitation-hardenable stainless steel, is also given in Table I, as well as the nominal composition of the materials used for a literature comparison. This material was received as solution-treated from 1800 F and was aged for 15 hr at 1325 F before machining and grinding. The finished specimens were heated for an additional hour at 1325 F to complete the aging treatment and to relieve machining and grinding stresses.

Two specimen designs were used in this study. The constant-stress specimen (Fig. 2) is designed so that the applied

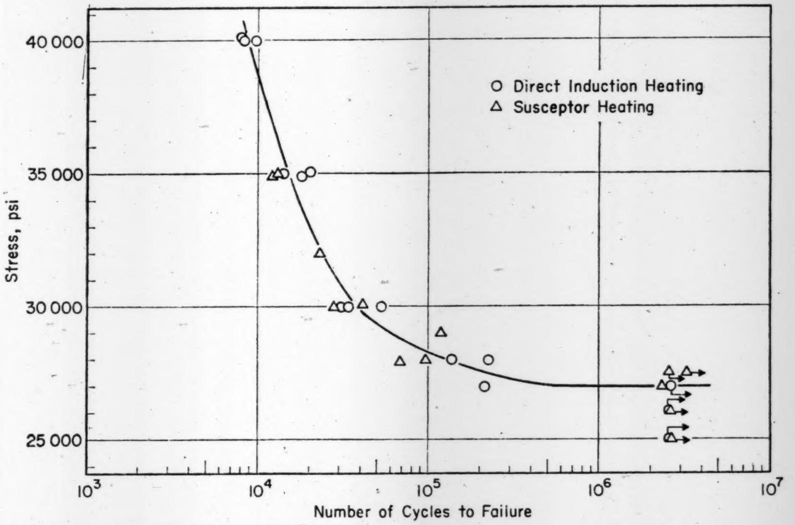

FIG. 4.—Fatigue Data for Type 304 Stainless Steel Showing Equivalence of Direct Induction Heating and Susceptor Heating.

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[3] H. C. Cross, "High-Temperature Tensile Creep, and Fatigue of Cast and Wrought High- and Low-Carbon 18Cr-8Ni Steel from Split Heats," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 56, p. 533 (1934).

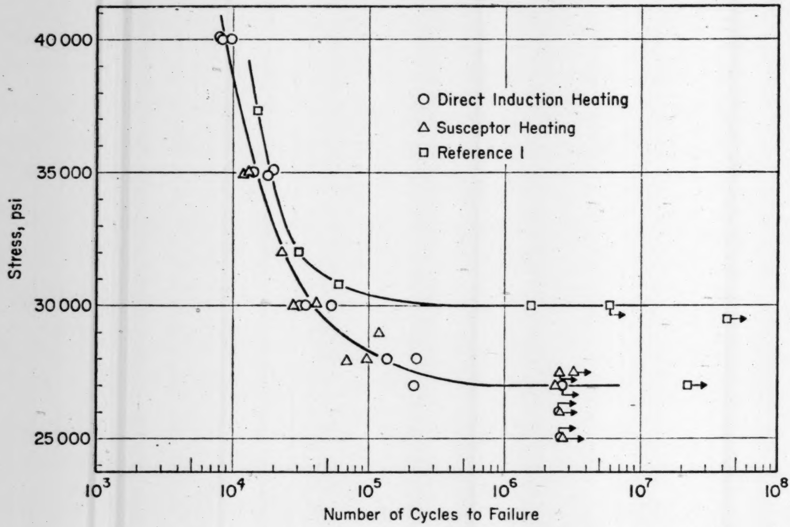

FIG. 5.—Comparison of Fatigue Data for Type 304 Stainless Steel at 1200 F.

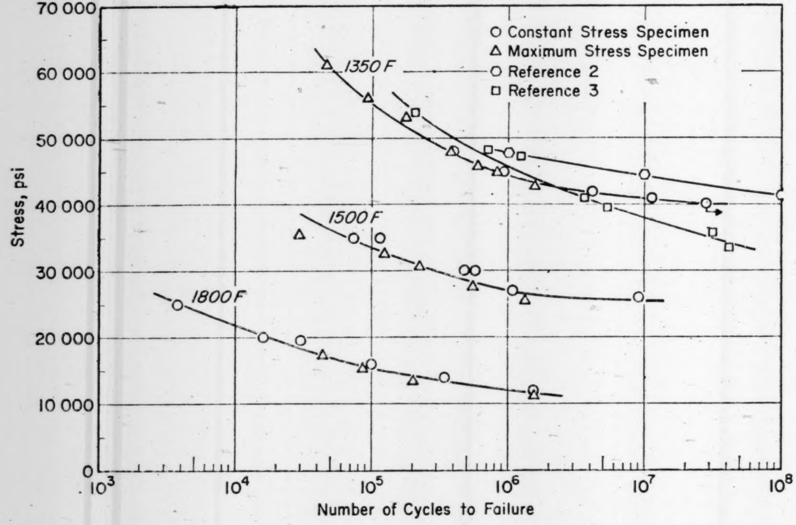

FIG. 6.—Fatigue Data for Type A-286 Stainless Steel Alloy at Various Temperatures: Comparison with Literature Data at 1350 F.

stress is constant over the tapered length; the applied stress is dependent on the applied load and the specimen thickness. In all cases, the stress reported is the calculated stress in the outermost fibers of the specimen according to elastic beam theory. The second type of specimen is a maximum-stress specimen (Fig. 3). In this case, the applied stress is dependent on the distance, L, between the applied load and the reduced section, the width of the reduced section, in addition to the specimen thickness, and the magnitude of the load. Whichever type specimen is used, it must be proportioned to attain desired stress levels with the 24-lb maximum load, without exceeding the maximum allowable deflection imposed by the coil.

Experience has favored the maximum-stress specimen in spite of a theoretical stress concentration caused by the reduced cross-section. Not only does it have greater flexibility of design, but it is also simpler to fabricate. In addition, it ensures failure at a fixed position.

RESULTS AND DISCUSSION

Influence of Direct Induction Heating:

Initially, there was some concern that induction heating would affect fatigue life. The possibility of such an influence lies in the nature of induction heating. Specifically, the initiation of fatigue cracks may interfere with the induced current flow in the fatigue specimen. Such an occurrence could cause overheating in the vicinity of the cracks, thus changing the rate of crack propagation and thereby fatigue life.

To investigate the possible effect of direct induction heating, type 304 stainless steel specimens were tested, using the susceptor coil described to avoid direct induction heating. In Fig. 4, these data are compared with data for specimens heated directly by induction. There is no apparent measurable difference between the two sets of data.

Comparison with Literature Data:

The literature[3] yielded furnace heating data for type 304 stainless steel at 1200 F (Fig. 5) that may be compared with data obtained in this study. A small difference between the two sets of data may reasonably be attributed to differences in material composition or in the type of fatigue testing unit used, or both.

An austenitic, precipitation-hardenable stainless steel, A-286, was tested at 1350, 1500, and 1800 F. Data available for comparison from the literature[4, 5] at 1350 F (Fig. 6) show a reasonable correlation between the three sets of data taken at 1350 F.

Figure 6 shows a rapid decrease in fatigue life with increasing temperature for this alloy; however there is no indication of a fatigue limit in the range of the data reported. Although both specimen designs were used in this phase of the testing program, there appears to be little difference in the results.

SUMMARY

A high-temperature fatigue unit utilizing induction heating has been developed and used successfully for high-temperature fatigue testing over a range of temperatures from 1200 to 1800 F.

The results of tests on type 304 and A-286 stainless steels are comparable to literature data available for these materials.

Special capabilities of the unit for high temperatures, thermal cycling, and environmental control appear possible with minor modifications.

Acknowledgment:

The Wiedemann Machine Co., Lepel High-Frequency Laboratories, Barber-Colman Co., Allegheny Ludlum Steel Corp., and Carpenter Steel Co. have contributed to this work financially and technically. The authors are particularly grateful for equipment and materials used in the investigation.

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[4] D. A. Roberts, D. B. Roach, and A. M. Hall, "Physical and Mechanical Properties of Nine Commercial Precipitation-Hardenable Stainless Steels," *DMIC Report No. 112*, Battelle Memorial Inst., Columbus, Ohio, May 1, 1959.

[5] "Elevated Temperature Fatigue Properties of Several High-Temperature Sheet Materials," Allegheny Ludlum Steel Corp. Research Laboratory, Watervliet, N. Y., Nov. 12, 1954, p. 4.

DISCUSSION

Mr. A. A. Mittenbergs.[1]—In a fatigue investigation[2,3] conducted a few years ago at Battelle Memorial Inst., induction heating was used to provide a temperature of 1800 F in molybdenum test specimens. These sheet specimens were subjected to an axial tension-tension loading. An induction coil of elongated shape having several windings was placed around the specimens for direct heating in a manner similar to that shown in Fig. 2 of the paper except that the specimens were in a vertical position.

In the early stages of our investigation, a problem of sparking between the coil and test specimen was encountered. The sparking occurred only at the start-up of the fatigue machine when it accelerated rapidly to the synchronous operating speed of 3600 cpm and passed through the natural frequency of the induction coil structure. This resulted in lateral motion between the coil and specimen which decreased the resisting air gap. For efficiency purposes, the coil was located rather close to the specimen (about 0.12 in.), but the amplitudes of the lateral vibrations were of small order and there was no metallic contact between the coil and the specimens. The problem was overcome by shutting off the induction furnace for a second or so at the critical moment. This experience, however, had indicated that relatively small lateral motion between the coil and specimen could cause sparking.

Because a lateral motion between the specimen and the coil is required in the investigation just described, I wonder (1) whether a similar sparking problem has been experienced during the development of the cantilever type bending test with direct induction heating, and (2) if such would be the case, how this problem was overcome.

In the investigation that I referred to earlier,[2,3] we were also very concerned with the possibility of an increase of temperature at the tip of the crack from induction heating. Because of the axial loading (rather than bending), we were in a better position to observe the cracks during their propagation with an optical pyrometer. We did not find any evidence of a temperature increase at the cracks.

Mr. J. A. Dunsby.[4]—The authors have drawn attention to the vexatious question of whether, with either induction or direct resistance heating, overheating at the tip of a crack will change the rate of crack propagation and hence reduce the apparent fatigue life. It appears from the paper, however, that the experiments using induction heating have to date been confined to unnotched specimens in which cracks would only be present for a relatively short proportion of the life. Thus any change in rate of crack propagation might be expected to have but secondary effect on life. With notched specimens, not only is the crack present for a longer proportion of the life, but the notch itself may give rise to temperature gradients.

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[1] Senior Program Director, Mechanical Engineering Department, Battelle Memorial Inst., Columbus, Ohio.

[2] A. A. Mittenbergs, D. N. Williams, R. I. Jaffee, H. J. Grover, and G. D. Haley, "Investigation of the Fatigue Properties of Molybdenum Under Various Conditions of Temperature, Coatings, and Stress Concentration," *WADD Technical Report 60-427* U. S. Air Force, 1960.

[3] A. A. Mittenbergs, G. D. Haley, and D. N. Williams, "Fatigue Properties of Uncoated and Coated Unalloyed Molybdenum at 1800 F, Room Temperature, and −40 F," *Proceedings*, Am. Soc. Testing Mats., Vol. 61, 1961, p. 757.

[4] Structures Laboratory, National Aeronautical Establishment, National Research Council, Ottawa, Ont., Canada.

While having the utmost sympathy with the authors on the subject of temperature control, it must be pointed out that the accuracy they have achieved is outside even the simple tension test requirements of ASTM Recommended Practice E 21.[5] This does not, however, appear to be reflected in the test results since the scatter is not excessive and the comparison with other published results, particularly in the case of the A-286, is most satisfactory. One is led to wonder on what basis temperature tolerances for fatigue testing should be established and whether, in fact, temperature tolerances that have been laid down for other forms of mechanical testing are excessively close.

MR. J. F. LIBSCH (*authors' closure by letter*).—Mr. Mittenbergs has described a problem of sparking between the induction coil and the fatigue specimen encountered in testing molybdenum at 1800 F. The authors experienced no such sparking with the equipment described. The tendency for sparking is increased as the electric potential between the coil and the specimen increases and as the air gap between the coil and specimen decreases. The arrangement described in this paper provides a large air gap. Proper coil design and grounding can reduce the potential.

It is gratifying to note that Mr. Mittenbergs did not find any evidence of a temperature increase at the tip of the propagating cracks in his work, thus confirming our observations. It is fortunate that the propagating crack, or a deliberate notch in a notched specimen, is so oriented with respect to the electromagnetic field established within the coil that localized or nonuniform heating is negligible. Our experience with the maximum stress specimens containing a notch, and with specimens showing a long life, lead us to believe that localized heating is truly negligible, and that conditions of testing which would increase the proportion of specimen life during which the crack was present would not significantly influence the results.

The reproducibility of results obtained with the equipment described by several separate investigators indicates an adequacy of temperature control, as pointed out by Mr. Dunsby. Nevertheless some refinements in instrumentation, including an electronic servomechanism recently added, offer promise of still more precise control.

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[5] Recommended Practice for Short-Time Elevated-Temperature Tension Tests of Materials (E 21), 1961 Book of ASTM Standards, Part 3.

IMPROVEMENT IN 7075-T651 ALUMINUM ALLOY FATIGUE LIFE BY COINING

By Richard E. Whaley[1]

Synopsis

Fatigue test results of aluminum tension specimens containing attachment holes and subjected to two methods of coining are compared to the results of a control group of uncoined specimens. The two methods of coining the test specimens were (1) surface working around the holes and (2) coining in the holes by using hardened steel dies. The tests show that the fatigue lives of coined specimens were increased over the fatigue lives of uncoined specimens for every die used. It is shown that coining both sides of $\frac{3}{8}$-in. thick aluminum specimens results in greater life improvement than that obtained by coining only one side. Results are presented which show increases in coined specimen life over uncoined specimen life of bolted joint specimens. Results are also presented which show life improvements for coined monoblock specimens loaded with an airplane maneuver load spectrum.

A prevailing problem in achieving structural integrity in aircraft is the reduction in fatigue life resulting from attachment holes. The cross-sectional strength of members is reduced not only by the material loss due to the hole, but by the stress concentration at the edge of the hole, which may be as high as three times the nominal stress in the cross-section. It is a well-known fact that increases in fatigue strength can be obtained by introducing residual compressive stresses by various techniques, such as cold working.

The object of the tests described in this paper was to increase the fatigue life of 7075-T651 aluminum alloy plate containing attachment holes by various techniques of coining around the holes with hardened steel dies. Constant level tests were conducted using conical shaped dies and surface-working ring-shaped dies on one surface and on both surfaces of the specimens. Constant level tests were also conducted on coined and uncoined bolted joint specimens. Tests were also conducted using an airplane maneuver load spectrum on coined and uncoined monoblock specimens.

Specimens and Specimen Preparation

All specimens were machined parallel to the rolling direction from the same lot of $\frac{3}{8}$-in. thick 7075-T651 aluminum alloy plate secured for fatigue test purposes. The average properties of six tension specimens machined from the plate were as follows: elongation in 2 in., 11.7 per cent; yield strength, 0.2 per cent offset, 73,100 psi; ultimate strength, 81,200 psi. After the specimen blanks were cut from the plate, the specimens were selected at random for machining and labeling. Specimens were 1.25 in. wide with a row of seven 0.313-in. holes at 1.25-in. spacing. This hole spacing of four diameters is usually the minimum spacing used in airplane construction. The theoretical stress concentration factor, K_t, for this configuration, calculated with the aid of data from Peterson,[2] is about 2.2. A typical specimen is shown in Fig. 1.

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[1] Structures Laboratory, McDonnell Aircraft Corp., St. Louis, Mo.

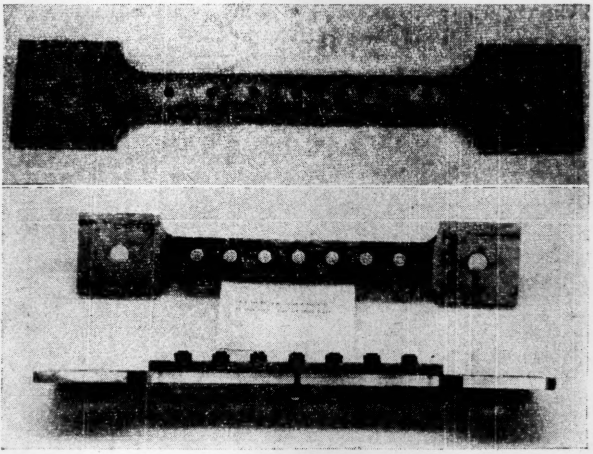

FIG. 1.—Typical Monoblock and Bolted Joint Configuration Tensile Fatigue Specimens.

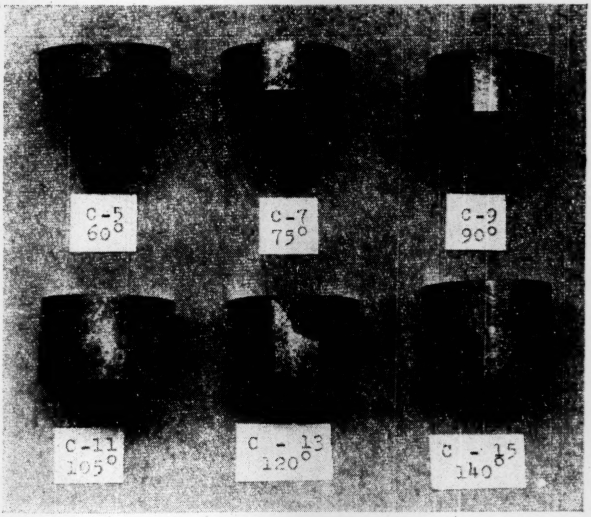

FIG. 2.—Cone-Shaped Dies.

Fifteen control (uncoined) specimens were tested. Two specimens were cut into halves at the center hole, and cold-rolled structural steel straps were bolted on to simulate bolted connections. Each specimen was match drilled with the straps, and holes were reamed so that a class 3 fit was obtained with NAS bolts. Each specimen half was labeled as a single specimen. After the first failure in each pair, the unfailed specimens were bolted together and testing

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[2] R. E. Peterson, *Stress Concentration Design Factors*, John Wiley and Sons, Inc., New York, N. Y., 1953, pp. 84, 94 (Catalog of Experimental and Theoretical Solutions).

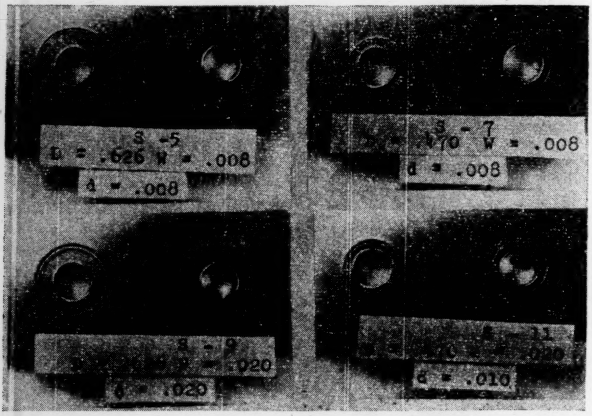

FIG. 3.—Typical Specimens Worked with Surface-Working Ring Dies Showing Diameter, Width and Depth of Groove.

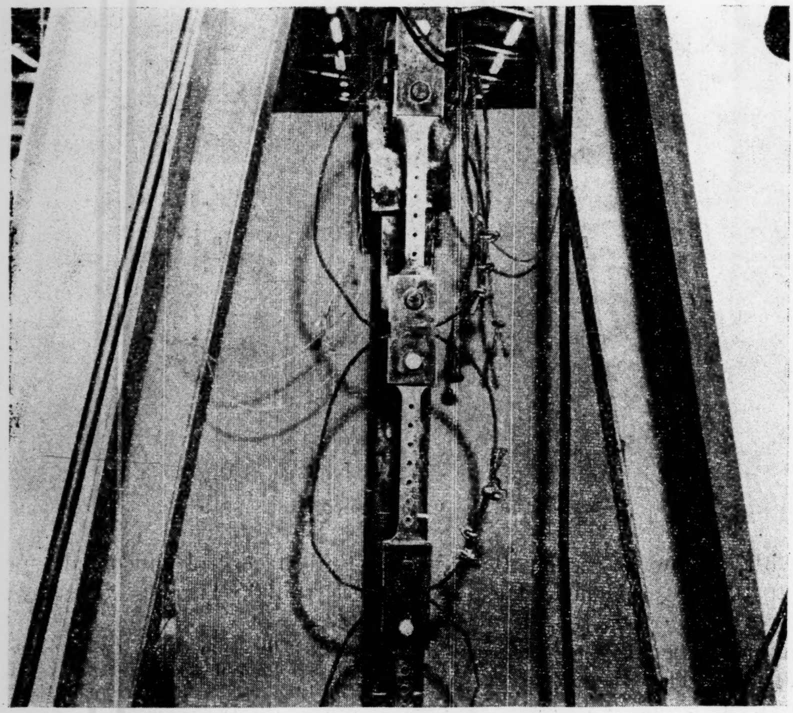

FIG. 4.—Coined Specimens Mounted in Tensile Fatigue Test Jig.

TABLE 1.—LOADING SPECTRUM.

Load[a]	Stress, psi	Cycles
35	14 700	1700
45	18 900	950
55	23 100	650
65	27 300	450
75	31 500	250
85	35 700	150
95	39 900	30
105	44 100	15
115	48 300	4
125	52 500	1.5
15.4	6 468	lower limit

[a] Load expressed in per cent of 42,000 psi used for constant level tests.

continued. The last specimen to fail was tested to failure using a dummy replacement. Figure 1 shows this bolted joint configuration.

Other specimens were coined with two types of dies. One type of die consisted of a cone which was pressed into the hole (Fig. 2). The angle of the cone is given for each die number in the photograph. The second type of die produced a circular groove in the specimen surface around the hole when the die was pressed against the specimen. Typical specimens worked with this type of surface-working ring die are shown in Fig. 3. The dimensions given in the photograph are the diameter, *D*, of the ring, width, *W*, of the groove, and depth, *d*, of the groove.

TABLE 2.—RESULTS OF SPECIMENS COINED WITH CONICAL DIES.

Specimen[a] Type	Mean Lifetime,[b, c] cycles	Life Increase[d]	Sample Standard Deviation of Log Lives	95 Per Cent Confidence Limits, cycles
Uncoined	5 985[e]	...	0.0681	4 740 to 7 550
SPECIMENS COINED ON ONE SIDE				
C-5	10 500	1.76	0.0071	10 085 to 10 935
C-7	9 040	1.51	0.0690	6 090 to 13 430
C-9	8 531	1.43	0.0841	5 265 to 13 825
C-11	8 690	1.45	0.0214	7 680 to 9 830
C-13	9 710	1.62	0.0516	7 220 to 13 550
C-15	10 020	1.68	0.0426	7 850 to 12 750
SPECIMENS COINED ON TWO SIDES				
C-5	14 610	2.22	0.1370	6 640 to 32 100
C-9	7 750	1.30	0.1005	4 355 to 13 800
C-13	19 900	3.33	0.0672	13 540 to 29 250
C-13[f]	9 720[g]	1.63	0.0396	8 215 to 11 500
C-13[f, h]	9 250	1.55	0.0084	8 815 to 9 700

[a] Specimens coined with 1400 lb unless otherwise noted.
[b] Three specimens tested unless otherwise noted.
[c] Value is the anti log of mean log lifetime.
[d] Life increase is ratio of life of coined specimens to life of uncoined specimens.
[e] Six specimens tested.
[f] Specimens reamed after coining.
[g] Four specimens tested.
[h] Specimens coined with 3000 lb.

Coined specimens were identified by a three-number code. The first number indicates the type of die used: S for surface working or ring die and C for cone-shaped die. The second number indicates the particular die of the type shown and labeled in Fig. 2, the third number is the specimen number of the series. Thus S-9-3 is a ring die surface-worked specimen with the dimensions shown in Fig. 3 and is the third specimen of the group.

TEST PROCEDURE

Specimens were bolted vertically in series and connected to hydraulic loading cylinders fastened to a structural jig (Fig. 4). As many as five specimens were installed in each string, and from one to four strings were tested simultaneously. Each string had a separate loading cylinder so that failure in one string did not affect the other strings. Failure is defined as complete specimen separation. All constant level specimens were cycled at about 40 cpm with a maximum stress of 42,000 psi. Cycling was conducted at a load ratio with $R = 0.10$. Several specimens were cycled with the airplane maneuver load spectrum shown in Table 1, which was applied repeatedly from the low loads to the high loads. Each application of the spectrum was called one program of loads. The spectrum shown in Table 1 was taken from Mil-A-8866.[3]

TABLE 3.—TEST RESULTS OF SPECIMENS COINED WITH SURFACE WORKING RING DIES.

Specimen[a] Type	Mean Lifetime,[b, c] cycles	Life Increase[d]	Sample Standard Deviation of Log Lives	95 Per Cent Confidence Limits, cycles	Depth of Die Impression, in.
MONOBLOCK SPECIMENS TESTED AT CONSTANT LEVEL					
Uncoined	5 985[e]	...	0.0681	4 740 to 7 550	...
S-5[f]	9 605	1.61	0.0725	6 335 to 14 560	0.005
S-7[f]	8 980	1.50	0.1270	4 335 to 18 610	0.004
S-9[f]	10 290	1.72	0.0298	8 670 to 12 210	0.020
S-9	23 100	3.87	0.0520	17 140 to 31 100	0.006
S-11	8 810	1.47	0.0341	7 250 to 10 710	0.011
S-17	21 150	3.54	0.1353	9 730 to 45 950	0.0025
S-17	32 150	5.38	0.0759	20 800 to 49 680	0.005
S-17	52 500	8.78	0.0373	42 400 to 65 040	0.014
S-17	39 250	6.57	0.1220	19 490 to 79 050	0.022
S-17	42 600	7.12	0.1128	22 350 to 81 180	0.011
S-19	19 800[g]	3.31	0.1279	11 530 to 34 000	0.005
S-19	57 100	9.55	0.0362	46 410 to 70 250	0.017
S-19	57 450	9.60	0.0785	36 610 to 90 110	0.023
S-19	57 850	9.66	0.1071	31 300 to 107 000	0.025
S-17 C-13[h]	23 800	3.98	0.2261	65 000 to 87 100	0.005
MONOBLOCK SPECIMENS TESTED WITH MANEUVER LOAD SPECTRUM					
Uncoined	122.3[g]		0.0685	91 to 163	
S-19	589.0[g]	4.80	0.0479	481 to 721	0.023
BOLTED JOINT SPECIMENS TESTED AT CONSTANT LEVEL					
Uncoined	8 000[g]		0.1336	4 545 to 14 065	
S-19	60 100[g]	7.50	0.1655	29 800 to 121 800	0.023

[a] Coined specimens worked on both sides unless otherwise noted.
[b] Three specimens tested unless otherwise noted.
[c] Life in programs for spectrum-loaded specimens.
[d] Life increase is ratio of life of coined specimens to life of uncoined specimens.
[e] Data repeated from Table 2.
[f] Specimens coined on one surface only.
[g] Four specimens tested.
[h] Specimens coined with conical dies and also worked with ring die.

RESULTS AND DISCUSSION

Results of tests on 106 specimens are summarized in Tables 2 and 3. It can be seen from the tables that an improvement in fatigue life was obtained with each die. For most configurations, three specimens were tested. It should be noted, however, that each specimen contained seven holes. Each mean lifetime, therefore, was lower than if each specimen could have been further tested so that failure occurred at every hole. The mean lifetime, and the scatter, were also less than those for an identical number of specimens each with a single hole having the same stress concentration. It is, therefore, difficult to analyze the data statistically because of the complex nature of the specimens and the small number of specimens of each configuration. Included in the tables, however, is the sample standard deviation of the log lives of each group. This sample standard deviation was used to calculate confidence limits of 0.95 for each group.

Conical Die-Worked Monoblock Specimens:

The 1400-lb load on the conical dies was selected as the maximum load that would permit insertion of a close tolerance bolt after coining. The deformation caused by the conical die-working operation is shown in Fig. 5. The deformation is local, and the grain structure appears to be affected only in the immediate vicinity of the deformation. It was not deemed practical to coin specimens with dies having angles less than 60 deg. Figure 5 shows that application of this steep-angle die caused deformation out from the surface as well as into the hole. Any decrease in cone angle would result in die contact, which could be produced better by working the inside of the holes with a different type of tool.

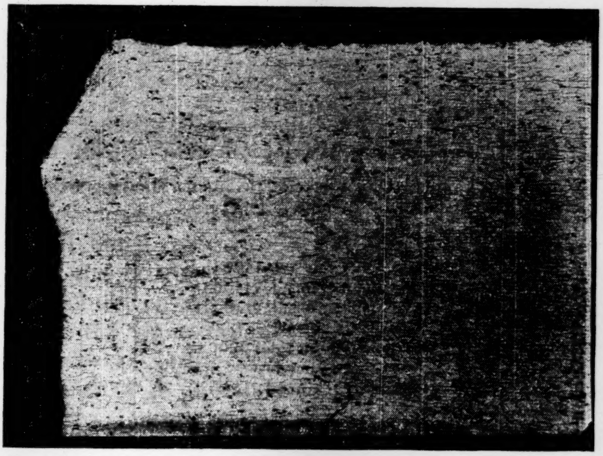

FIG. 5.—Hole in 7075-T651 Aluminum Alloy Coined with 60-deg Conical Die; Etched with Kellers Etch (×100).

For the specimens coined on one surface only, the point of crack initiation was near the coined surface in at least one half of the specimens. Figure 6 shows specimens with beach marks, indicating crack initiation, near both the coined and uncoined surface and near the center. This phenomenon

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[3] "Airplane Strength and Rigidity Reliability Requirements, Repeated Loads, and Fatigue," Military Specification Mil-A-8866, May, 1960.

indicates that this type of coining would not have a large effect on the fatigue life.

Results of specimens coined with the conical dies are shown in Fig. 7. There is a modest increase in fatigue life over uncoined specimens with the 90-deg conical die and greater increases as the angle of the cone becomes smaller or larger. The increase reaches a peak at about a 120-deg angle and then drops off rapidly. The curve for specimens coined on one side has the same shape as the curve for specimens coined on both sides, but the increase in life over uncoined specimens is not nearly as much. The effects of this type of coining on one surface or both may vary with specimen thickness. The results obtained apply only to the thickness of the aluminum specimens tested. The fatigue life of specimens coined on one side was

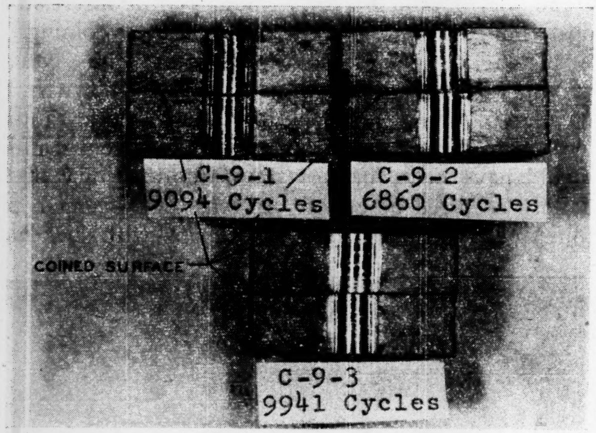

FIG. 6.—Failed Surfaces of Conical Die-Worked Specimens Showing Beach Marks Near Coined and Uncoined Surface and Near Center of Specimen Thickness.

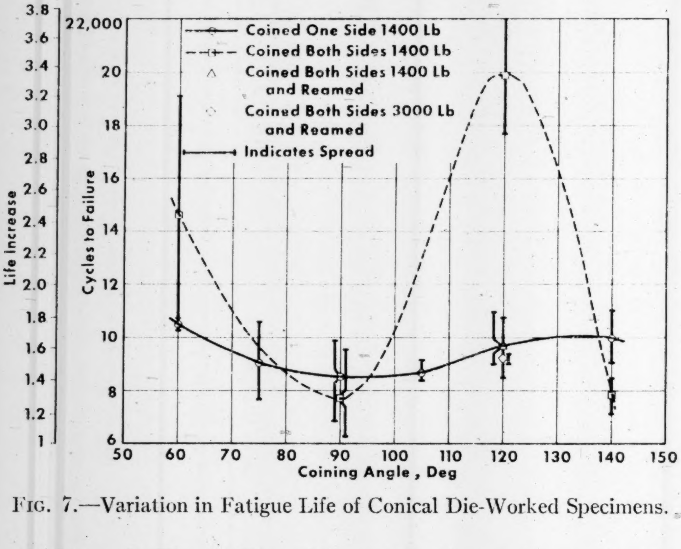

FIG. 7.—Variation in Fatigue Life of Conical Die-Worked Specimens.

always less than twice the life of uncoined specimens, while the life increase for specimens coined on both sides with the 120-deg die was about 3.3 times the life of uncoined specimens. A slight change in cone angle from this optimum condition, however, results in a large decrease in fatigue life. The reason for this large change in lifetime may be due to the small number of specimens tested. The confidence limits listed in Table 2 indicate that the curves shown could be considerably modified by larger numbers of specimens for each coining operation. The larger mean lifetimes of the specimens coined on one side over those of specimens coined on both sides with conical dies of 90 and 140 deg may also be due to the small number of specimens tested.

Reaming the holes to remove only the bulged-out area of specimens coined on both sides with the 120-deg conical die resulted in reducing the fatigue life to that of specimens coined on one side only. Approximately the same fatigue life was achieved when specimens were coined with a force of 3000 lb and then reamed. The results are summarized in Table 2.

Ring Die-Worked Monoblock Specimens:

Four dies (S-5 through S-11) were used initially on one surface of the specimens to determine which type of configuration had the best potential for fatigue life improvement. The load on the dies was selected in such a way that the depth of penetration would equal the width of the groove. The force on the S-11 die, however, was reduced so that the holes in the specimen would not be deformed. The result was a depth of only half the groove width. It was not deemed practical to increase the groove depth beyond the groove width. Dies of this configuration would be difficult to fabricate, would be easily subject to damage, and would tend to cut into the specimen rather than coin the specimens. The actual groove depths (listed in Table 3) obtained with the S-5 and S-7 dies were less than the values listed in Fig. 2 because of springback. Figure 2 and Table 3 indicate the use of the two dies with ring diameters twice the hole diameter resulted in greater life increases than obtained with dies having rings 1.5 times the hole diameter. The specimens with the wider groove (worked with the S-9 die) had a larger life increase than those with the narrower groove (worked with the S-5 die). The lower penetration with the S-11 die accounts for these specimens having the smallest increase in fatigue life over uncoined specimens. It is difficult to determine the values of all variables for optimum life improvement, as is pointed out by Phillips.[4] It

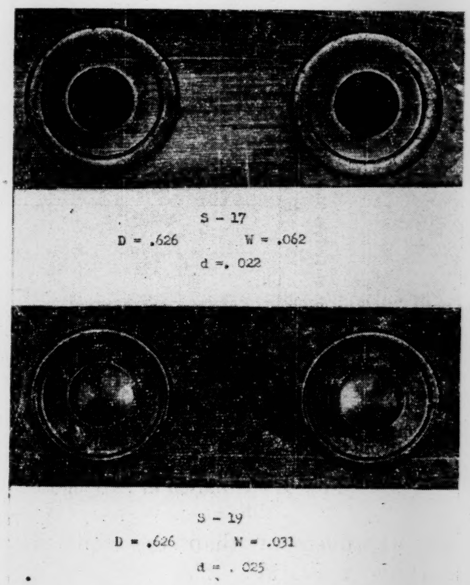

FIG. 8.—Typical Specimens Worked with Surface-Working Ring Dies Showing Diameter, Width, and Depth of Groove.

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[4] A. Phillips, "Improvement of Fatigue Life of Aircraft Components by Coining," ASME Paper No. 61-AV-35, March, 1961.

is believed, however, that a further increase in groove diameter would result in interaction of the stresses around the grooves of adjacent holes. This belief is based on stresses shown by Phillips.[4] No attempt was made to determine residual stresses in any of the specimens for the data of Tables 2 and 3.

All specimens coined on one surface only had beach marks that indicated crack initiation at or near the opposite surface to the coined surface. This indicates that surface working with ring dies has a larger potential for fatigue life improvement than the cone die-working operation. The S-9 die was then applied to both surfaces of three specimens, and the life increased to about 3.9 times the life of uncoined specimens (as shown in Table 3). The crack initiation for the three specimens was near the center of the specimen thickness. Phillips[4] showed that coining both surfaces of relatively thin sheet resulted in internal shear cracking and better results were obtained with coining only one side. The surface working process affects both surfaces, depending upon specimen thickness. No attempt was made to evaluate this variable and the results are only valid for the thickness of aluminum tested.

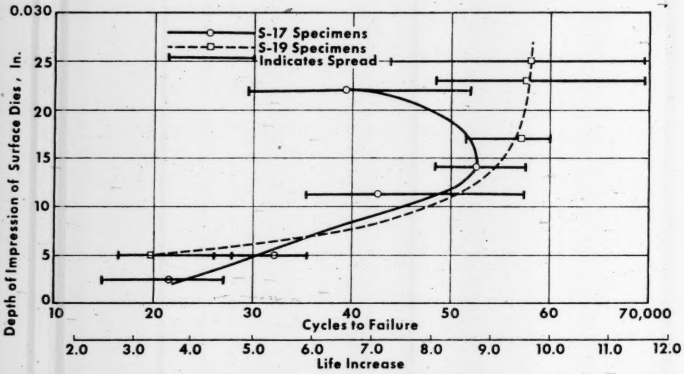

FIG. 9.—Variation in Fatigue Life of Ring Die Surface-Worked Specimens.

Further testing was performed by use of two more dies with ring diameters twice the hole diameters and wider width rings than those of the S-9 die. Specimens coined with each of these dies are shown in Fig. 8. The widths of the grooves are indicated on the photographs. Results of tests of these specimens are shown in Fig. 9. With penetrations much lower than obtained with the S-9 die, the specimen fatigue life was more than three times the life of uncoined specimens. This fatigue life was comparable to the life obtained with the S-9 die. Both the S-17 and S-19 dies at these penetrations were deforming the hole in the specimen very slightly. With increasing penetration, the specimen life increased to almost ten times the life of uncoined specimens.

Lifetimes of specimens with the S-17 die reached a peak and then decreased with increasing penetration. One contributing factor to this decrease was that the working of the S-17 die to a depth of 0.022 in. was so severe that the sides of the specimen were bulged out. Lifetimes of specimens with the S-19 die reached an optimum value which was higher than that when the S-17 die was used. No bulging of the sides of these specimens (with the S-19 die)

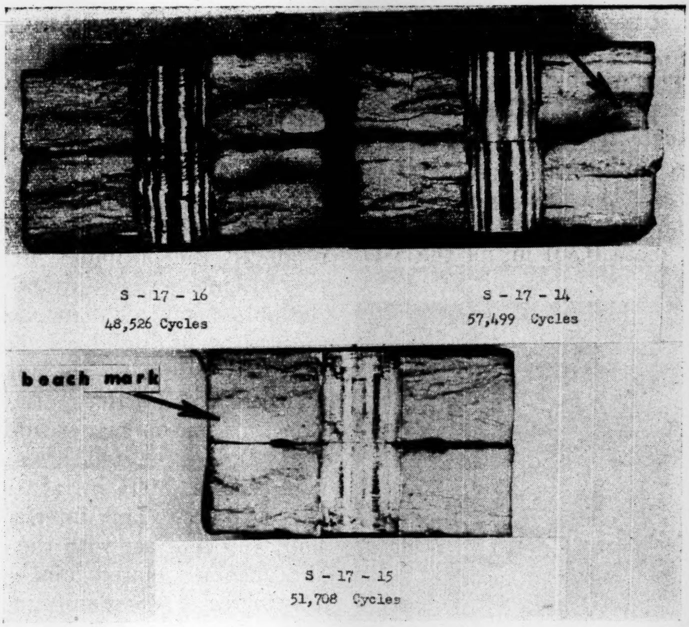
S - 17 - 16
48,526 Cycles
S - 17 - 14
57,499 Cycles
beach mark
S - 17 - 15
51,708 Cycles

FIG. 10.—Failed Surfaces of High Lifetime Ring Die Surface-Worked Specimens Showing Beach Marks at Edge of Specimens.

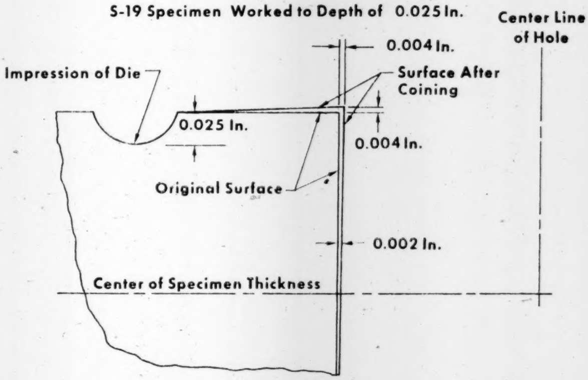
S-19 Specimen Worked to Depth of 0.025 In.
Center Line of Hole
0.004 In.
Impression of Die
Surface After Coining
0.025 In.
0.004 In.
Original Surface
0.002 In.
Center of Specimen Thickness

FIG. 11.—Deformation of Ring Die Surface-Worked Specimen.

was evident because the groove made by the S-19 die was narrower than that made by the S-17 die.

The point of crack initiation for the high lifetime specimens was not at the hole. Figure 10 shows typical specimen beach marks which indicate crack initiation at or near the edge of the specimen. There was only one exception for specimens having lives greater than 36,000 cycles. This point of crack initiation away from the hole, and therefore away from the coining, is another indication that higher penetrations would not result in increased fatigue life.

Deformation of Specimens:

Measurements of specimens coined

to the greatest penetration with each of the two dies, S-19 and S-17, showed that the surface was bulged out 0.004 to 0.005 in., respectively. This bulge increased gradually from zero at the groove to maximum at the edge of the hole as shown in Fig. 11. The holes at the surface of the plate were deformed to a diameter of about 0.301 in. for the S-17 specimens, and to 0.305 in. for the S-19 specimens, from the original diameter of 0.313 in. The centers of the holes were deformed only about half as much as that at the edge of the plate. These deformations are not local in nature as deformations with the conical die-worked specimens were, because the point of contact of the die is one-half a hole diameter from the edge of the hole. It is therefore believed that reaming of the holes back to 0.313 in. would not have too great an effect on the increased fatigue life. The deformations were measured from the photomicrographs and no attempt was made to determine residual stresses.

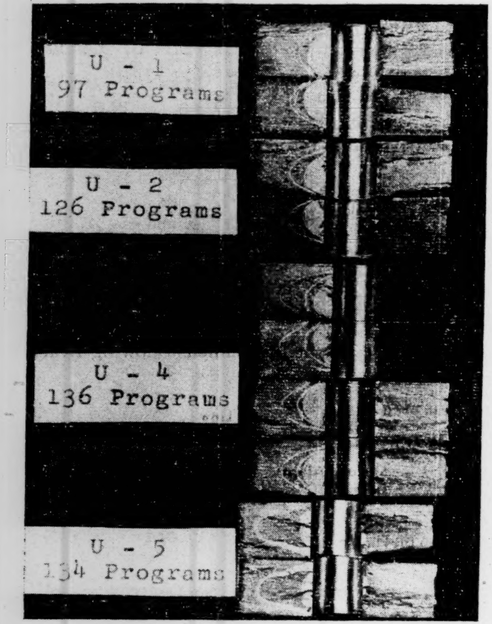

FIG. 12.—Failed Surfaces of Spectrum-Loaded Uncoined Specimens Showing Beach Marks.

Bolted Joint Configuration:

Two more specimens were coined with the S-19 die to a depth (0.023 in.) that produced the optimum lifetime. These two specimens were cut and mounted in the bolted joint configuration. The procedure for testing these specimens was identical to that for the uncoined specimens. Because of the specimen deformation due to coining, an interference fit of 0.007 in. existed at the specimen surface and 0.003 in. at the center of the thickness. This interference of the bolt is in contrast with the class 3 fit of the uncoined bolted joint specimens. A life increase was determined with the realization that the increase could vary considerably depending on the difference of class of fit between the uncoined specimens and the coined specimens. The resultant fatigue life of these specimens, listed in Table 3, showed a factor of 7.5 times the fatigue life of uncoined bolted joint specimens. This 7.5 factor increase was slightly less than the 9.6 factor increase of the monoblock configuration. Beach marks on these bolted joint specimens indicated that the point of crack initiation was still at the hole. It is conceivable that different penetrations or a different class of fit would result in a further increase in fatigue life of this configuration, which would equal or exceed the 9.6 factor increase of the monoblock configuration.

Combination Ring Die-Worked and Conical Die-Worked Specimens:

Three specimens were coined with the S-17 die on both surfaces to a depth of 0.005 in., and the 120-deg cone angle die was then used on both surfaces. The results of tests on these specimens (presented in Table 3) showed an increase in fatigue life (over uncoined specimens) greater than that obtained with the C-13 conical die alone, but was less than that obtained with the S-17 ring die alone. It does not appear practical, therefore, to use the conical dies in conjunction with the ring-type dies.

S - 19 - 22
500.0 Programs
S - 19 - 23
636.0 Programs
S - 19 - 24
614.9 Programs
S - 19 - 25
615.0 Programs

FIG. 13.—Failed Surfaces of Spectrum-Loaded Ring Die Surface-Worked Specimens Showing Beach Marks.

Spectrum-Loaded Specimens:

Uncoined specimens U-1 through U-5 loaded with the spectrum shown in Table 1 had a mean life of 122.3 programs. The failed surfaces are shown in Fig. 12. Specimen U-4 had a crack in one other hole, and the area on the opposite side from the crack was sawed through to show the beach marks on the cracked surface. The beach marks on all specimens were very clear and contained concentric rings. It is conceivable that each ring represents one program of 4200 cycles but is was difficult to count the rings close to the crack initiation point.

The last few rings on each specimen have larger areas between them than between the first rings. This area may be the result of cracking at the high loads in the spectrum. This cracking was followed by the low loads of the next program which started another ring. All specimens, with the exception of U-5, failed on the last load of a program; U-5 failed in the middle of a program. The beach marks on U-5 indicate that one load, probably the last load in the spectrum prior to failure, cracked most of one side of the specimen. The last ring was then produced by the first half of the last program.

Four more specimens were coined with the S-19 die to a depth which resulted in the optimum life increase during the constant level tests (9.6 times uncoined specimen lifetime). These specimens were loaded with the spectrum shown in Table 1 and had a fatigue life 4.8 times the life of the uncoined specimens. It was expected that these specimens would have a life increase (over uncoined specimen life) at least as great as a factor of 9.6, which was the increase for the same configuration tests at constant level. This estimate was based on results obtained by Phillips,[4] who showed increases in fatigue life for similar tests for the complete *S-N* curve. In these tests the endurance limit had been raised, and life increases were thus greater for stresses approaching the endurance limit than for low lifetime specimens. The linear cumulative damage hypothesis would therefore predict a life increase equal to or greater than the 9.6 factor obtained at high constant level.

The beach marks on the specimens shown in Fig. 13 indicated that the point of crack initiation was at the hole. This crack initiation area was in agreement with the crack initiation area of specimens tested at constant level in which the life increases (over uncoined specimens) were of the same magnitude. This crack initiation area was not in agreement, however, with that of specimens coined to the same depth with the same die and tested at constant level. Two of the four specimens had beach marks which were much smaller than on any other specimens tested. Calculations of the static strengths of these cracked specimens, based on data by McEvily, Illg, and Hardrath,[5] could not justify the failure of S-19-22, which had the smallest beach mark. The notch strength of this specimen was calculated to be in excess of the highest load in the spectrum, while the specimen failed during the second lowest load level in the spectrum. Calculations of the static strength of several other cracked specimens, based on data by McEvily, Illg, and Hardrath,[5] were very close to the loads at which they failed during the fatigue tests. No explanation for the premature failure of specimen S-19-22 can be found.

CONCLUSIONS

The results of tests on 7075-T651 aluminum alloy tension specimens containing attachment holes, to evaluate methods of coining to increase the fatigue life, support the following conclusions:

1. Any coining with either a cone-shaped die or a surface working ring-type die tended to increase specimen fatigue life.
2. Coining only one side of a $\frac{3}{8}$-in. thick plate specimen resulted in a moderate increase in specimen fatigue life, while greater increases were generally obtained by coining both sides.
3. Coining specimens with a cone-shaped die resulted in better specimen fatigue life increases with a cone angle of 120 deg than with any other angle tested. Slight changes from this angle in each direction resulted in much smaller increases in specimen fatigue life. This trend, however, is based on a very small number of specimens.
4. Reaming the deformed area from conical die-worked specimens resulted in a large loss of the originally improved fatigue life.
5. Coining with ring-type dies produced better fatigue life when the ring was twice the hole diameter rather than 1.5 times the hole diameter. Better fatigue life increases were obtained when the width of the ring was increased.
6. Fatigue life increased rapidly with increasing depth of penetration of a ring-type die but tended to reach an optimum when the initiation of the crack moved from the area of the hole (or coined area) to the edge of the plate. Fatigue life began to decrease if the die began to bulge the sides of the specimens.
7. Bolted joint configuration specimens coined with ring-type dies had slightly less fatigue life improvement (over uncoined specimen fatigue life)

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[5] Arthur J. McEvily, Jr., Walter Illg, and Herbert F. Hardrath, "Static Strength of Aluminum-Alloy Specimens Containing Fatigue Cracks," *NACA TN3816*, Oct., 1956.

than did the monoblock specimens coined with ring-type dies. The class of fit of the coined specimens, however, was considerably different from that of the uncoined specimens. The difference in class of fit variable would affect the life increase. The point of crack initiation remained at the hole in the long lifetime configuration.

8. Specimens coined with both a conical die and a ring-type had fatigue life increases (over uncoined specimen fatigue life) which were not as great as the life increase for specimens coined with the ring-type die alone. The increase, however, was better than that obtained with the conical die alone.

9. Specimens tested with an airplane maneuver load spectrum did not exhibit as large an increase in fatigue life (over uncoined specimen fatigue life) as did a similar configuration tested at high constant level. The life increase for spectrum-loaded specimens was 4.8 times the life of uncoined specimens, as compared to 9.6 times the life of uncoined specimens for specimens tested at high constant level.

10. The point of crack initiation for the spectrum-loaded specimens, coined with the ring die to the optimum depth, was at the hole. This crack initiation point was similar to that for specimens tested at high constant level and with about the same life increase, but unlike the crack initiation point for the optimum depth configuration tested at high constant level.

DISCUSSION

MR. THOMAS ZAJAC.[1]—The techniques described are interesting and appear to have merit from the standpoint of introducing a controlled and reproducible amount of cold working at holes in structural members. Two questions are raised relative to the data presented.

The method employing a cone shows a large increase in life for a 120-deg cone angle versus other cone angles. Is there a rational explanation for this result?

MR. RICHARD E. WHALEY (*author*).—The larger increase in life for the 120-deg cone angle was probably magnified as a result of the small number of samples. Statistical analysis indicates that the life difference might not be as great for a larger number of samples. The larger life increase, however, may be attributed to a larger volume of cold worked material. The point of die contact at the specimen surface around the hole, coupled with the vertical direction of applied load, indicates that the most cold worked volume would depend on the angle of die contact. No attempt has been made, however, to actually calculate the critical angle.

MR. ZAJAC.—Coining one side of the hole indicates a significant life increase. If the opposite side is unaffected, coining one side, in effect, reduces the volume of non-cold-worked material in each specimen. Is the life increase compatible with the reduction in material volume? If not, are there other contributing factors involved in the life increase, or is this simply a manifestation of small sample size?

MR. WHALEY.—Coining only one side of a specimen does not result in a completely unaffected opposite side. A more important factor than volume reduction is the residual compressive stress. The residual stress probably diminishes to a minimum value at the opposite surface from the coined surface. The life increase depends on the residual stress magnitude and also the stress gradient in the coined volume.

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[1] Head, Materials and Standards, Hamilton Standard, Division of United Aircraft Corp., Windsor Locks, Conn.

AN EVALUATION OF THE INFLUENCE OF CYCLIC PRESTRESSING ON FATIGUE LIMIT*

By G. W. Brown[1] and C. E. Work[2]

Synopsis

The fatigue limits of SAE 4340 steel for 16 combinations of cycle ratio and prestress level were evaluated using the up-and-down or staircase method. Cycle ratios of 5, 15, 35, and 70 per cent at prestresses 12.5, 25, 37.5, and 50 per cent above the fatigue limit of the virgin material were used and approximately 15 specimens were tested for each condition. The fatigue limit was found, in general, to be reduced by prestressing, and it was found possible to express the fatigue limit of the prestressed specimens as a definite function of prestress amplitude and cycle ratio. Limited results available from three other references were found to correlate with the behavior observed in this study by accounting for differences in the yield strength of the material.

A great deal of attention has been given in recent years to predicting fatigue life of members in which the stress amplitude fluctuates in a regular or even a random manner. Theories such as those by Miner (**1**),[3] Corten (**2**), and others for rationalizing the cumulative amount by which various numbers of cycles of stress affect the subsequent fatigue life are expressed in one of two ways. They are expressed either in terms of total expected cycles to failure under the varying pattern of stress amplitude or in terms of life at some subsequent constant test stress amplitude after a period of prestressing of whatever nature is being evaluated. Most of these theories are set up in such a way that they can shed no light on how the various stress histories influence the fatigue limit, if one exists.

Some attention has been given, however, to accounting for fatigue damage in terms of alterations of both finite fatigue life and fatigue limit. Henry (**3**) proposed a simple equation which he applied with some success to data from earlier work by Bennett (**4**) and Kommers (**5**). His theory employed a simplified expression for the *S-N* curve and appeared to match the data for fatigue life more closely than for fatigue limit. Manson and his associates have proposed a theory (**6**) which involves the entire *S-N* curve and uses certain assumptions about the knee of the curve and a point high in the finite life zone.

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* Presented at the Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] Research Engineer, Aluminum Laboratories Ltd., Kingston, Ontario, Canada; formerly associated with the Michigan College of Mining and Technology, Houghton, Mich.

[2] Professor and Head, Department of Engineering Mechanics, Michigan College of Mining and Technology, Houghton, Mich.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper.

Certain early damage studies used the fatigue limit to judge whether damage had occurred and in some instances evaluated damage in terms of change in fatigue limit (4,5,7). By their very nature, coaxing studies have involved progressively elevating the fatigue limit as well as drastically increasing the life at stresses moderately above the fatigue limit.

The study described in this paper is a quantitative evaluation of the magnitude of change in fatigue limit that results from various cycle ratios of prestress at various levels above the fatigue limit. It is broader than any of the earlier studies cited in covering a more complete set of combinations of prestress level and cycle ratio. Erickson's earlier work (8) with the same material indicated how the various prestress histories influence the finite life portion of the *S-N* diagram. The prolonged work required to study the influence on the fatigue limit, which was not complete when reference (8) was published, is described here.

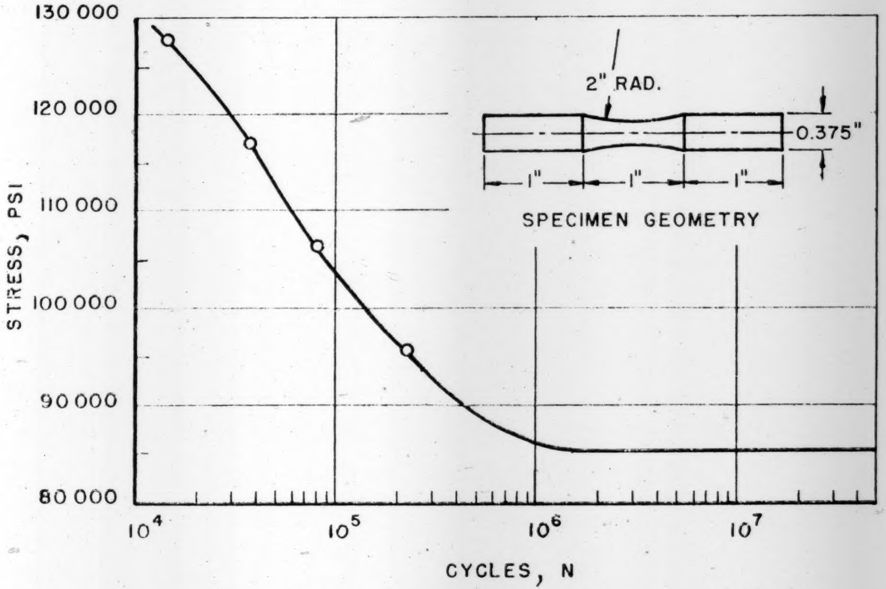

FIG. 1.—Basic *S-N* Diagram for SAE 4340 Steel.

EXPERIMENTS

The material used in this investigation was SAE 4340 steel, heat treated to a Rockwell hardness, C scale, of 27 to 30, a static tensile strength of 156,000 psi, and a yield point of 145,000 psi. Fatigue tests were carried out in Krouse high-speed rotating-cantilever-beam machines with unnotched specimens approximately $\frac{1}{4}$ in. in diameter at the test section. The material and test procedure are specified in more detail in reference (8).

The fatigue limit of the virgin material was determined with 20 specimens to be 85,300 psi by the up-and-down or staircase method described in the Appendix.[4] The finite-life portion of the *S-N* curve for the virgin material was determined by testing 15 specimens to failure at each of four levels of 50, 37.5, 25, and 12.5 per cent above the fatigue limit. The *S-N* diagram for the virgin material is given in Fig. 1.

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[4] See p. 711.

TABLE I.—FATIGUE LIMITS AFTER PRESTRESSING.

$\frac{\sigma_p}{\sigma_e}$, per cent	σ_e, psi, at			
	C = 5 per cent	*C* = 15 per cent	*C* = 35 per cent	*C* = 70 per cent
150.....	77 500	74 100	62 500	52 900
137.5...	81 100	70 300	64 300	43 900
125.....	82 300	75 100	68 500	51 700
112.5...	82 500	82 900	76 900	64 900

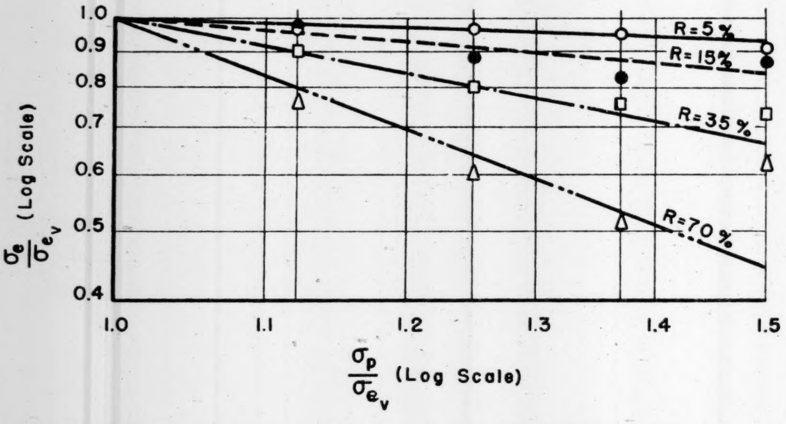

FIG. 2.—Influence of Prestress on Fatigue Limit of SAE 4340 Steel Specimens.

To produce the alterations of the material that result from cycles of prestress, approximately 60 specimens were subjected to similar prestressing at each test stress level, and approximately 15 of these were cycled for 5 per cent cycle ratio, C. Other groups of approximately 15 specimens each were subjected to cycle ratios of 15, 35, and 70 per cent at each of the four levels. All cycle ratios for prestressing were based on the number of cycles corresponding to the mean of the logarithms of life of virgin specimens cycled to failure at the corresponding stress level.

The extent to which prestressing influenced the fatigue limit was then evaluated by applying the staircase method to determine a new fatigue limit for each group of specimens. All specimens being prestressed to cycle ratios of 5, 15, and 35 per cent survived the prestressing and were retested at another level. However, a total of seven specimens failed before reaching the 70 per cent prestress-cycle ratio. None of these prestress failures occurred at the highest prestress level. Wherever failure occurred during prestressing, calculations are based on surviving specimens, as the staircase method requires calculations based on the smaller group. In these cases the survivors were the smaller group.

TEST RESULTS

The fatigue limits after prestressing in the experiments described are listed in Table I. Each value listed represents the median obtained by the staircase method. The effect of prestressing was pronounced and fairly consistent (Fig. 2).

Stresses, represented by the coordinates of each point in Fig. 2, are expressed in nondimensional form, each stress value being divided by 85,300 psi, the fatigue limit of the virgin material, σ_{e_v}, and are plotted to a logarithmic scale. The ordinates represent the median fatigue limits σ_e obtained experimentally by the staircase method and abscissas represent the amplitude of prestress, σ_p. The trends in the results are approximated by a family of straight lines for different values of cycle ratio, C. The slope, α, of each of these lines is related to cycle ratio in a remarkably orderly manner as shown in Fig. 3. Thus, with a few notable exceptions, the influence of prestress on fatigue limit observed can be expressed by the simple exponential equation:

$$\frac{\sigma_e}{\sigma_{e_v}} = \left(\frac{\sigma_p}{\sigma_{e_v}}\right)^{-\alpha} \quad \ldots\ldots\ldots\ldots (1)$$

where $\alpha = \beta C$ and $\beta = 2.86$ for the present study. The constant β is an empirical coefficient and in this paper will be called the cycle ratio coefficient.

Relevant data from various other studies have been evaluated in the same manner. Most of the other investigators studied only one prestress amplitude, and fatigue limits were evaluated with fewer specimens than were used in the present research program. In spite of various possible sources of differences, it is interesting to observe that their results can be expressed by the same exponential equation (Eq. 1) but with a different cycle ratio coefficient β to account for the different materials, all of which were steel.

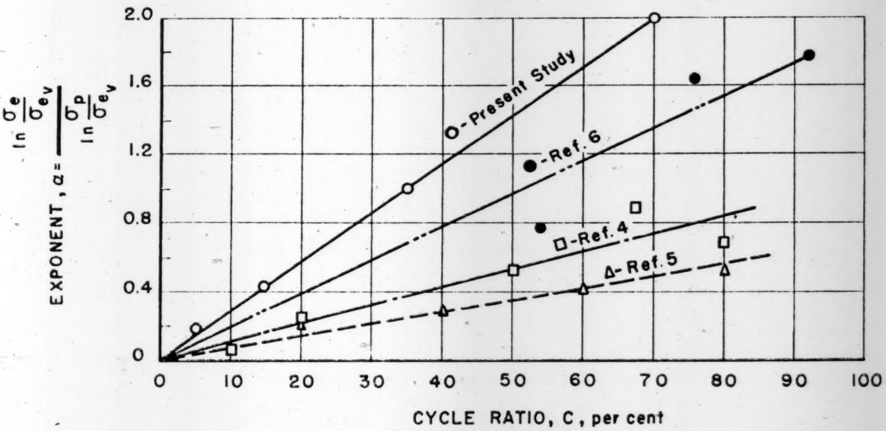

FIG. 3.—Influence of Cycle Ratio on the Reduction of Fatigue Limit by Prestressing.

For the data summarized in Table II, there is a consistent tendency for higher strength materials to have larger values of the cycle ratio coefficient, β obtained from slopes in Fig. 3. This trend appears as a straight line in Fig. 4 where β is plotted to a logarithmic scale versus the yield strength. Yield strength was chosen as the measure of the static strength for the abscissa, since using this property resulted in a more linear graph than when ultimate strength was used. From this graph, an empirical relation can be deduced

$$\beta = \phi e^{\theta \sigma_y} \quad \ldots\ldots\ldots\ldots (2)$$

where $\phi = 0.457$ and $\theta = 1.238 \times 10^{-5}$. This is an empirical relation based on the present tests plus data from three other papers in which values relevant to this discussion were discovered. Certain limited counter tendencies which may be quite valid have been ignored here which, along with differences in specimen geometry and methods of performing and analyzing tests, would make extensive generalization of these trends unwise.

TABLE II.—COMPARISON OF FATIGUE DAMAGE EXPONENTS FROM DIFFERENT INVESTIGATIONS

Author and Reference	Steel	Static Strength, psi		Specimen	β
		σ_y	σ_u		
Kommers (**5**)	SAE1020	39 020	58 750	smooth	0.72
Bennett (**4**)	X 4130	62 500	104 200	notched	1.01
Manson et al (**6**)	SAE4130	120 000	133 800	smooth	1.96
Brown and Work (present paper)	SAE4340	145 000	156 000	smooth	2.86

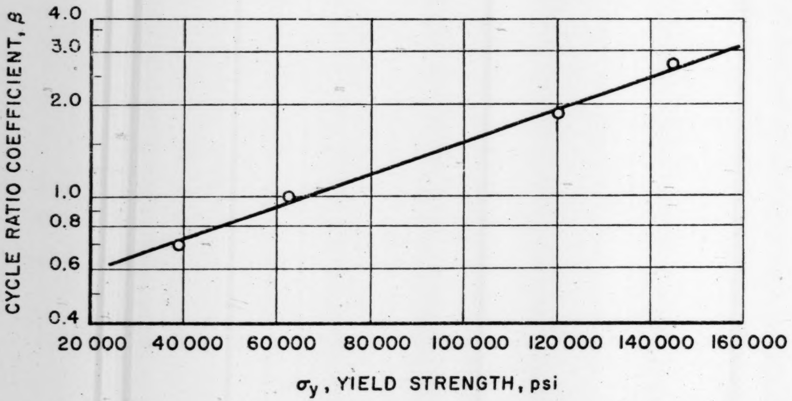

FIG. 4.—Variation of Cycle Ratio Coefficient with Yield Strength of Material.

In spite of the various possible limitations, the simplicity of the relation presented in Eq 2 makes it interesting. The authors make no claim, at this point, to a rational development of this equation; however, the acceptance of this relation as an approximate experimental fact makes it possible to introduce the strength of the material into Eq 1 presented earlier. Thus the fatigue limit for prestressed specimens is obtained as a rather simple function of prestress amplitude, cycle ratio, and the static yield strength of the material, namely,

$$\sigma_e = f(\sigma_p , C, \sigma_y) \dots\dots\dots\dots (3)$$

CONCLUSIONS

The results of this investigation show that prestressing caused a consistent reduction in fatigue limit of the steel considered. The fatigue limit after prestressing could be expressed approximately by the equation

$$\frac{\sigma_e}{\sigma_{e_v}} = \left(\frac{\sigma_p}{\sigma_{e_v}}\right)^{-\beta C}$$

where:

σ_e = fatigue limit after prestressing,
σ_p = prestress amplitude,
σ_{e_v} = fatigue limit of virgin material,
β = cycle ratio coefficient, a material constant, and
C = cycle ratio of prestress.

Limited data available in the literature from three other investigations were found to correlate well with this observation, and it was found that, although the cycle ratio coefficient was different for the different steels, it was an exponential function of yield strength:

$$\beta = \phi e^{\theta \sigma_y}$$

where ϕ and θ were constants.

Therefore, it was concluded that the endurance limit of prestressed specimens was a definite function of the virgin endurance limit, prestress amplitude, cycle ratio, and yield strength of the steel.

Acknowledgment:

This investigation was conducted in the research laboratories of the Department of Engineering Mechanics of the Michigan College of Mining and Technology. The authors are especially indebted to the contributions of T. Dimoff and W. H. Erickson to the early stages of the project on which this paper is based.

APPENDIX

THE STAIRCASE OR UP-AND-DOWN TEST METHOD

In the staircase or up-and-down method, a sequence of testing is prescribed (**9**) which is intended to concentrate observations about the median fatigue limit. The first specimen is tested at a stress chosen to be as close as possible to the anticipated median fatigue limit. This first guess may be based on prior knowledge or on preliminary tests. If the first specimen fails, a second is tested at a stress one step lower. If it does not fail, the second test is at a stress one step higher. After the second test, further tests are carried out, each test at a stress one step lower or higher than the one immediately preceding it, lower if the preceding specimen failed, higher if the preceding one survived. Some arbitrary number of cycles is chosen as the basis for assuming survival. In the present investigation specimens that had not failed by 10^7 cycles were called survivors.

The optimum step size is equal to one standard deviation, σ, of the distribution of fatigue limits, but the standard deviation is not known until all tests have been completed. Since the step size must be chosen in advance, it must be estimated. The standard deviation, σ, has been found to vary with the type of steel within the range of 1500 to 5000 psi, and it is believed that steps of any size from $\sigma/2$ to 2σ will give satisfactory results. Since the steel used in this investigation had medium strength, a step size of 3000 psi was chosen. The estimated standard deviation from the tests to determine the virgin fatigue limit by the method described below was 3250 psi, and those determined for prestressed specimens provided further verification that 3000 psi was an appropriate step size.

The average of the values of stress for all specimens tested in this sequence is an estimate of the median fatigue limit. In this research, the procedure of Dixon and Mood (**9**) for estimating the median was used in which the analysis of results is based on the group (survivors or failures) with the smallest number. Specimens used in the initial probing to select an appropriate stress at which to begin the sequence are omitted from the calculations. N represents the total number of one particular event; thus N_f is the total number of failures and N_s the total number of survivors.

$$N = \sum_{i=0}^{i=k} n_0 + n_1 + n_2 + \cdots + n_k$$

If the analysis is based on failures, the quantities n_0, n_1, n_2 above represent the number of failures at each stress level where n_0 is the number of failures at the lowest stress level at which failure occurs.

The estimated median fatigue limit, m, is then given by:

$$m = y' + d\left(\frac{A}{N} - \frac{1}{2}\right) \ldots \quad \ldots (4)$$

where y' is the stress corresponding to the lowest stress at which failures occur, d is the step size, and A is the first moment given by the expression:

$$A = \sum_{i=0}^{i=k} in_i = 0 \times n_0 + 1 \times n_1 + 2 \times n_2 + \cdots + kn_k$$

The standard deviation of the endurance limits of individual specimens is estimated by s where

$$s = 1.62d\left(\frac{NB - A^2}{N^2} + 0.029\right) \ldots (5)$$

where B is the second moment given by

$$B = \sum_{i=0}^{i=k} i^2 n_i = 0^2 \times n_0 + 1^2 \times n_1 + 2^2 \times n_2 + \cdots + k^2 \times n_k$$

This method of estimating the standard deviation is useful for values of $(NB - A^2)/N^2$ greater than 0.3 but breaks down for values less than 0.3.

If the calculations are based on survivors, the expressions for A and B are the same and N is N_s. N_s is determined in the same way as N_f except that N_s is the summation for values of i associated with survivors. The value for m is obtained by use of Eq 4 except that the minus sign is replaced by a plus sign. The stress with which n_0 is associated is not different when calculations are based on survivors.

The procedure of Dixon and Mood varies slightly from this for calculations based on survivors. However, it was found that by using the method outlined above, when $N_f = N_s$, the values obtained for m are identical, as would be expected.

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(7) H. J. French, "Fatigue and Hardening of Steels," *Transactions*, Am. Soc. for Steel Treating, Vol. 21, p. 899 (1933).

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DISCUSSION

Mr. S. S. Manson.[1]—The authors are to be commended for their very interesting and useful treatment of an important subject. The determination of the remaining endurance limit after prior stressing is perhaps one of the more valuable aspects in the study of cumulative fatigue damage study. Their extremely simple formula which does not involve measurements other than those normally made in a fatigue test constitutes a valuable contribution.

TABLE III.—REMAINING ENDURANCE LIMITS OF PRESTRESSED MATERIALS IN ROTATING BENDING.

Material	Yield Strength, psi	Prestress Level, psi	Prestress Cycle Ratio, n/N	Original Material Endurance Limit, psi	Experimental Endurance Limit After Prestress, psi	Predicted Endurance Limit After Prestress, psi
AISI 52100	260 000	160 000	0.7	104 000	94 000	3 140
			0.8		86 000	1 850
5456-H311 aluminum	34 000	45 000	0.67	22 000	10 000	15 800
			0.80		7 000	14 800
304 stainless steel	108 000	120 000	0.70	75 000	42 000	42 000
AISI 4130	120 000	100 000	0.50	70 000	55 000	51 800
			0.70		39 000	45 500
			0.85		39 000	41 200

Research on the determination of remaining endurance limit after prior fatigue has been under way at the writer's laboratory for a number of years, and it is interesting to compare the results of tests conducted to date with predictions by the authors' method. The results are shown in Table III for four materials, together with the yield stress, prestress level, cycle ratio, initial endurance limit, remaining endurance limit, and predicted endurance limit based on the formula in the report. It is seen that very good predictions are obtained for the stainless steel and the 4130 steel. For the aluminum alloy, the predictions are unconservative—that is, they predict remaining endurance limits that are higher than the observed values. For the very hard steel, 52100, the predictions are overconservative. For example, at a cycle ratio of 0.7, a predicted endurance limit of 3140 psi compares to the measured value of 94,000 psi, and at a cycle ratio of 0.8 the predicted endurance limit of 1850 psi compares to an experimentally determined endurance limit of 86,000 psi. It is particularly interesting that this very high-strength steel does not, therefore, suffer as high a loss of endurance limit as predicted by the formula in this report. The reason for this result is currently under investigation.

Mr. J. A. Bennett[2] (*presented in*

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[1] Chief, Materials and Structures Division, National Aeronautics and Space Administration, Lewis Research Center, Cleveland, Ohio.

[2] Metallurgist, National Bureau of Standards, Washington, D. C.

written form).—The authors interpret their data to mean that there is a simple relation between the prestress amplitude, the cycle ratio, and the resulting fatigue limit. It is unfortunate that in selecting data from reference (4) of their paper for evaluation by their formula they chose only one of the four sets of values available. The other three show, I believe, that the relation is not simple but is almost discontinuous. The staircase method of determining the fatigue limit tends to obscure this discontinuity.

The accompanying Fig. 5, plotted from data in reference (4) of the paper, shows on the left the effect of cycle ratio on the fatigue limit of smooth specimens. The basis for calculating cycle ratio in this investigation was N_c, the number of cycles to start of crack propagation, in contrast to Brown and Work's values which were based on the number of cycles to fracture. Even at a cycle ratio of 90 per cent, the fatigue limit had dropped less than 10 per cent, and there was little difference in the effect of prestressing at the two stress levels. On the right, I have attempted to show the relation between the area of the propagating fatigue crack and the fatigue limit. The curve must go through zero at 100 per cent crack area, and one other estimate of its location is available. This is the fatigue limit, from Fig. 10 of reference (4), of notched specimens that had been cracked approximately 17 per cent by prior fatigue stressing. The stress concentrating effect of a crack in a smooth specimen would be greater than in the notched one, so the dashed curve is drawn below this point. If the relation between number of cycles and fatigue limit were continuous, these two curves should extrapolate to the same point, since 100 per cent damage corresponds to the start of crack growth. Despite the meager data for the right-hand graph, it seems clear that this is not the case and that there must be a very rapid drop in the fatigue limit just prior to the development of a visible crack.

Because of the probable existence of this drop, it was unfortunate that the authors chose the staircase method of evaluating the fatigue limit, as it would concentrate the tests at stress levels intermediate between the cracked and uncracked fatigue limits. Thus the result obtained depends only on the proportion of each type of specimen resulting from a given prestress and does not give any information about the true shape of the curve.

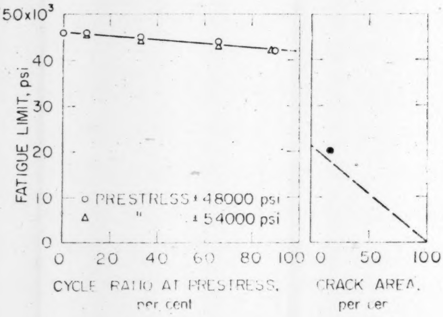

FIG. 5.—Evidence of a Discontinuity in the Relation Between Prestress Cycles and Fatigue Limit; Plotted from Data in Reference (4) of the Paper.

In view of the above comments, I would like to ask the authors if they made any effort to determine if the specimens contained detectable cracks after prestressing? Also was any attempt made to analyze the fatigue life data for those specimens that broke in the staircase tests?

MR. H. O. FUCHS[3] (*by letter*).—This paper establishes some important and neatly stated conclusions by an extensive series of carefully conducted tests. It seems likely that the results will be used by future investigators and designers;

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[3] University of California, Department of Engineering, Los Angeles, Calif.

therefore it is important to remember that these conclusions, which are derived from tests with fully reversed loading (zero mean stress), apply only to fully reversed cycles. They are in part contrary to what we know about the influence of prestressing in fluctuating loading.

Springs and gears are typical of machine elements subject to fluctuating loads. In the manufacture of springs it is common practice to "preset" them by a few cycles of overload.[4] This invariably improves the fatigue performance. If the presetting is mistakenly done in the direction apposed to the applied load, the fatigue performance is damaged.[5]

We can explain this by the influence of mean stress on permissible stress range: In any machine element some areas (the surface) are more vulnerable to fatigue damage than others. By a few cycles of high load—even below the static yield strength—these areas are made to yield. The well-known "settling" of springs in service is evidence of this. The highly stressed yielded areas then develop residual stresses opposite in sign to the load stress. The stress range is shifted from the original (for example, zero to 100 per cent) to a new (for example, −20 per cent to +80 per cent) which gives longer life or a higher endurance load stress. The effect is especially strong in the presence of sharp stress concentrations.[6]

The conclusions of the paper apply to the fatigue limit in fully reversed loading and perhaps also to some other conditions of loading. They do not apply to the fatigue limit in general.

For conditions of substantial mean stress the fatigue limit can be and often is raised by prestressing to a high overload.

It seems quite unlikely to the writer that there is any simple general law of cumulative fatigue damage. The cumulative damage probably depends on mean stress and on stress concentration as much as on material and cycle ratio. In this view, the present paper is an excellent investigation into cumulative damage under one special set of conditions.

MR. C. E. WORK (*authors' closure by letter*).—The authors are indebted to Mr. Manson for making available additional results from his research to check further the usefulness and limitations of the equations presented in the paper and to Mr. Bennett for calling attention to the data in reference **(4)** for unnotched specimens. In reference **(4)** Mr. Bennett had so neatly tabulated the effect of prestressing on endurance limit of *notched* specimens (Table V, reference **(4)**) that the corresponding data for unnotched specimens (which were not tabulated) had been overlooked by the authors. Only the data for prestressing at stresses above the fatigue limit was relevant to this study.

Most of the data presented by Manson are in reasonable agreement with the empirical relationships presented in the paper, the values for type 304 stainless steel coinciding exactly with predictions. However, predictions for AISI 52100 are clearly inaccurate. This fact, along with Bennett's data for unnotched specimens, tends to underscore the authors' statement in the paper that, although there is some evidence to support Eq 2, extensive generalization of this empirical relation would be unwise.

Mr. Fuchs' experience with very high unidirectional prestressing helps define further the bounds of usefulness of the authors' equations. It should be pointed out that nominal stresses reported in the paper were well within the proportional-elastic range. The fact that inelastic processing results in a different type of behavior for large mean stresses is not, in itself, evidence that the trends reported for rotating-beam tests would necessarily be limited to zero mean stress.

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[5] N. E. Hendrickson, "Special Techniques for Increasing Strength and Fatigue Life of Steel Stressed in Torsion," *ASTM Bulletin*, No. 224, Sept., 1957, p. 40.

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No cracks were observed after prestressing except in specimens which were considered to have failed. No attempt has yet been made to analyze fatigue life data from specimens which failed in staircase tests.

A STUDY OF FATIGUE LIFE UNDER RANDOM LOADING*

By Herbert A. Leybold[1] and Eugene C. Naumann[1]

Synopsis

Simplified load schedules based on statistics obtained from several different methods of analyzing a random-load time history having the properties of band-limited white noise were used to conduct programmed axial-load fatigue tests on 2024-T3 aluminum-alloy edge-notched sheet specimens. Additional tests were conducted using load schedules which varied randomly. A comparison of the test results is made to determine whether or not the fatigue-inducing characteristics of the given random time history are retained when simplified load schedules are used.

An analytical method was developed to generate, digitally, random time histories having arbitrarily shaped power spectra. A programmed axial-load fatigue machine has also been developed to conduct tests based on the statistics obtained from the generated time histories. The machine uses an IBM punch card system to apply up to 55 discrete load levels in any given sequence.

Results indicate that a considerable variation in life is obtained in tests scheduled according to statistics of a given random time history analyzed according to several different counting methods.

The aircraft designer is acutely aware of the structural fatigue problems existing in modern aircraft. In order to design a structure having a reasonable fatigue life he must be able to estimate its life under service loading. Since there are no known theories that can be used to predict accurately the life under service loading, the designer must rely on the fatigue test as a means of obtaining a reliable estimate of life. If the random-load time histories encountered in service could be duplicated in a laboratory test, the estimate obtained from such a test would undoubtedly be considered reliable. Existing fatigue testing equipment is usually limited to applying simple cyclic loads; therefore the designer must estimate service life from simple laboratory tests. Several counting methods have been devised for reducing the service loading history to numerical form. These reductions result in several different sets of load statistics that can be used to program fatigue tests which simulate, with varying degrees of complexity, the service loading history. The purpose of the present investigation is to determine whether or not the programmed fatigue test, based on the load statistics obtained from the various counting methods, adequately retain the significant fatigue-inducing characteristics of the service loading history.

To determine the significant fatigue-inducing parameters of any random-load time history (not necessarily the service loading history), an analytical method was developed to generate, with the aid of a digital computer, random time histories having arbitrarily shaped power spectra. A digital rather than an analog type of representation was chosen for the following two reasons: (1) the digital time history is in a form directly amenable to reduction by various counting methods, and (2) the statistics obtained by the various counting methods can be punched on IBM cards and used as the input to a programmed axial-load fatigue testing machine.

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* Presented at the Sixty-sixth Annual Meeting of the Society.

[1] Aerospace Technologist, NASA Langley Research Center, Langley Station, Hampton, Va.

This testing machine was developed in order to conduct tests using the load statistics obtained from any digitally generated time history. The machine is capable of applying loads in either a random or programmed sequence.

To limit the scope of this paper, only one time history having the properties of band-limited white noise was generated and analyzed according to several different counting methods. The load statistics obtained from these counting methods were used to conduct programmed axial-load fatigue tests on 2024-T3 aluminum-alloy edge-notched sheet specimens having a theoretical elastic stress concentration factor of 4. Fatigue life estimates obtained from these tests were compared with estimates obtained from tests conducted using a load schedule which varied randomly. In this manner, it was possible to determine which programmed tests preserved the significant fatigue-inducing characteristics of the generated random time history.

GENERATION OF A RANDOM TIME HISTORY

Random numbers were generated, normally distributed, and numerically filtered to obtain a random time history having the properties of band-limited white noise. Of all generators tested, the random number generator selected had the least amount of correlation between numbers. Each random number was considered to be a sample of a continuous random time history taken at discrete uniform intervals of time. For convenience, the time history was normalized to have a unit variance. The following is a description of the procedure used to simulate digitally a random time history having the properties of band-limited white noise. The procedure to be described can be used equally well for digitally simulating other random time histories having arbitrarily shaped power spectra (**1**).[2]

Random Number Generator:

Random numbers were obtained, using standard digital computer techniques, with a fixed-point pseudo random number generator developed at the National Bureau of Standards (**2**). Each generated random number, R_N, was obtained from the previous random number R_{N-1} by taking the last eleven digits of the product R_0R_{N-1} where $R_0 = 5^{15}$ and $N = 1, 2, 3, \cdots$. Numbers were then selected at random from the generated R_N. Only the first six digits, p, of the randomly selected eleven-digit number were used in this investigation. Approximately 320,000 random numbers were obtained in this manner, each having an equally likely chance of occurring (that is, uniform probability distribution) where $0 \leqq p \leqq 999999$.

Transformation to Normal Distribution:

The random numbers were transformed into a normal distribution with mean equal to zero and unit variance by an approximate formula developed by Tukey (**3**). This was done in order to simulate a stationary, Gaussian random process. The equation required that the random numbers, p, be between zero and one; therefore all numbers, p, were divided by 10^6 and designated q. The expression is

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

$$X = 4.91[q^{0.14} - (1 - q)^{0.14}]$$

where q is the normalized random number and X is the normal deviate. In the present investigation it was found that when X became greater than 2.4 there were significant departures from the normal distribution. Hence it was necessary to make an additional correction to the equation as follows:

$$X \approx X' = 4.91[q^{0.14} - (1 - q)^{0.14}]$$

$$X = X' \quad \text{when} \quad |X'| \leqq 2.4$$

$$X = |X'| + 0.13\,(|X'| - 2.4)^2$$

$$\text{when} \quad |X'| > 2.4$$

where the sign of X takes the sign of X'. It should be noted that all normal deviates fall in the range $-4.91 \leqq X \leqq 4.91$.

It is assumed that the normally distributed random numbers represent a sampling of a continuous time history, $y(t)$, at discrete uniform intervals of time $\Delta t = \frac{1}{2}$ sec. Consequently, the random numbers can be thought of as a discrete set of values y_i equal to $y(t)$ when $t = i\,\Delta t$ and undefined in between. There is no loss of information when making the above assumption if $y(t)$ contains no frequencies greater than the Nyquist or folding frequency, f_F (4), where

$$f_F = \frac{1}{2\Delta t}$$

If frequencies higher than f_F are present in the data they will contribute energy or power to the lower frequencies which will result in errors in the power spectrum. In order to avoid this situation, the higher frequencies should be removed by appropriate filtering.

It is also assumed that the time history, $y(t)$, is stationary (that is, statistical properties are invariant with time) and Gaussian in nature.

Numerical Filtering of Random Numbers:

If the random process $y(t)$ is passed through a linear system having an amplitude or frequency response, $F(f/f_F)$, a new random process, $y(t)$, will be obtained. The frequency response $F(f/f_F)$ can be defined such that the new random process, $Y(t)$, will have a specified power spectrum, provided that $y(t)$ is stationary and Gaussian and the system is linear. This can be done by using the input-output relation of power spectral analysis which states that the product of the input power spectrum $\varphi_{\text{in}}(f)$ and the square of the absolute value of the frequency response $|F(f/f_F)|^2$ (also called a transfer function) is equal to the output power spectrum $\varphi_{\text{out}}(f)$. Thus

$$\varphi_{\text{out}}(f) = \left| F\left(\frac{f}{f_F}\right) \right|^2 \varphi_{\text{in}}(f) \ldots\ldots (1)$$

The frequency response, $F(f/f_F)$, can be determined from this equation since $\varphi_{\text{in}}(f)$ can be calculated (using the relationship between the autocorrelation function and power spectral density (4), and $\varphi_{\text{out}}(f)$ is the specified power spectrum, in this case band-limited white noise. Knowing $F(f/f_F)$, the new time history, $Y(t)$, can now be calculated using the following equation:

$$Y(t) = \int_{-\infty}^{\infty} h(\tau)\, y(t - \tau)\, d\tau \ldots\ldots (2)$$

where $h(\tau)$ is the response of the linear system to a unit impulse and is the Fourier integral of the frequency response. Conversely, the frequency response is the Fourier transform of the impulse response.

In order to solve Eq 2 numerically, the original time history, $y(t)$, is replaced by a discrete set of values y_{i+K} and the impulse response by a set of filter factors a_K. The new time history, $Y(t)$, having the desired shaped power spectrum is represented by a discrete set of values, Y_i, which are determined as follows:

$$Y_i = \sum_{K=-M}^{M} a_K y_{i+K}$$

Details for computing the filter factors, a_K, obtained by representing the frequency response function with a Fourier cosine series, are given in Appendix I.[3] A sample of the filtered time history obtained in this manner is shown in Fig. 1. For clarity, the values Y_i have not been plotted but rather the faired curve through these values. The input-output relationship used to obtain this time history is also shown in Fig. 1.

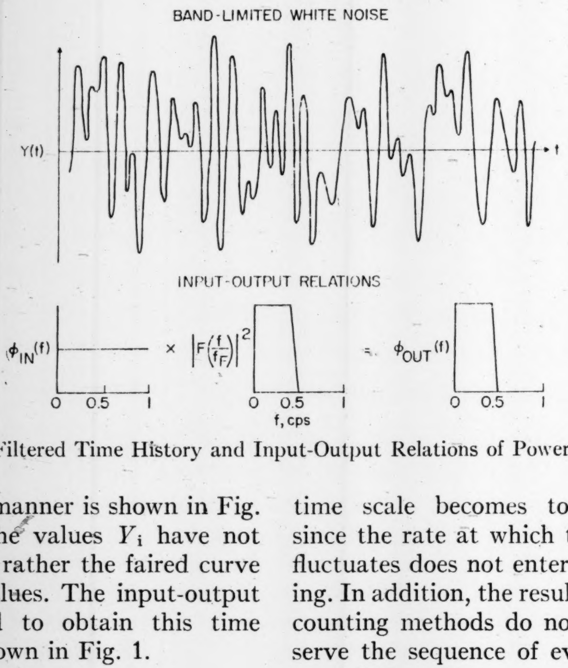

FIG. 1.—Sample of Filtered Time History and Input-Output Relations of Power Spectral Analysis.

Analysis of Random Time History:

The digitally generated random time history described in the previous paragraphs has been analyzed according to several different counting methods. The results obtained are several sets of statistics which describe, with varying degrees of complexity, the fluctuations encountered in the generated time history. The statistics are obtained by counting the number of times a certain event occurs throughout the random time history. An event may be considered to be a peak value (positive or negative), a distance between peak values (a range), or the crossing of a particular level by the time history in either the positive or negative direction. Events are counted in each of 50 equal intervals over the range −5.0 to +5.0 which encompasses the maximum and minimum values of the random numbers generated. The time scale becomes totally irrelevant since the rate at which the time history fluctuates does not enter into the counting. In addition, the results of the various counting methods do not normally preserve the sequence of events. However, special procedures were employed to retain the sequence for three conditions in this investigation.

The counting procedures to be used in this investigation have been previously discussed by Schijve (5). A brief description of each counting method used plus some of the counting results on a time history having the properties of band-limited white noise and consisting of

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[3] See p. 730.

320,000 random numbers are presented in the following paragraphs.

Peak Count Method.—This method provides a count of all the positive and negative peak values occurring in each of the 50 intervals of $Y(t)$. The peak values counted are indicated in the upper portion of Fig. 2. Also presented in this figure are the frequency distributions of the positive and negative peaks. Peaks counted in a given interval of $Y(t)$ as indicated in the figure are plotted at the center of the interval. It should be noted that the distributions of positive and negative peaks are symmetrical about zero, which is a good indication that the generated time history is truly random. Another method of counting the peaks is to count only the positive peaks above zero and negative peaks below zero. The results for this latter method of counting can be obtained from Fig. 2 by eliminating the positive peaks occurring below zero and the negative peaks occurring above zero. A modification of the latter method is frequently used for counting maneuver loads encountered in a service load time history of a fighter-type aircraft.

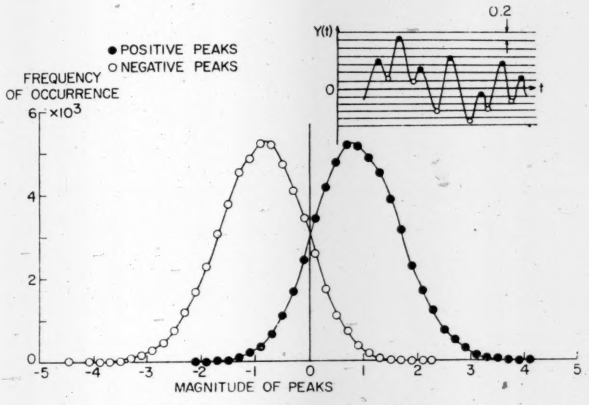

FIG. 2.—Distributions of Positive and Negative Peaks. Band-limited white noise consisting of 320,000 random numbers.

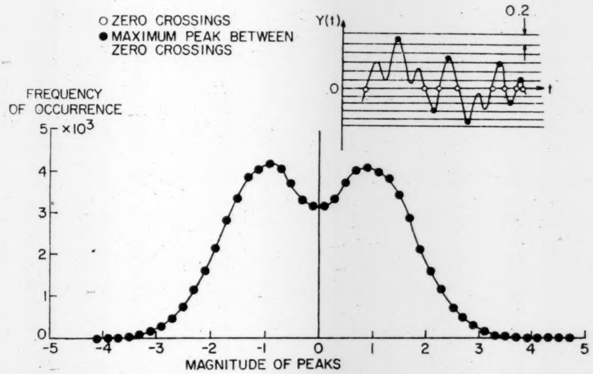

FIG. 3.—Distribution of Maximum Positive and Negative Peaks Between Zero Crossings. Band-limited white noise consisting of 320,000 random numbers.

Maximum Peaks Between Zero Cross-

ings.—As indicated in Fig. 3, only the highest peaks between zero crossings are counted. This counting procedure eliminates fluctuations of considerable size, as can be seen by comparing the frequency distribution of the maximum peaks between zero crossings (Fig. 3) with the distribution of all the peaks (Fig. 2). This method is similar to that generally used for counting gust loads encountered in a service load time history of a transport-type aircraft.

Amplitudes Regardless of Mean.—An amplitude is defined as half the range between successive peaks. It is considered to be positive when the slope of the curve between successive peaks is positive and negative when the slope is negative. This counting method gives no information about the peak values. The values counted are indicated in Fig. 4 with the results of the counting.

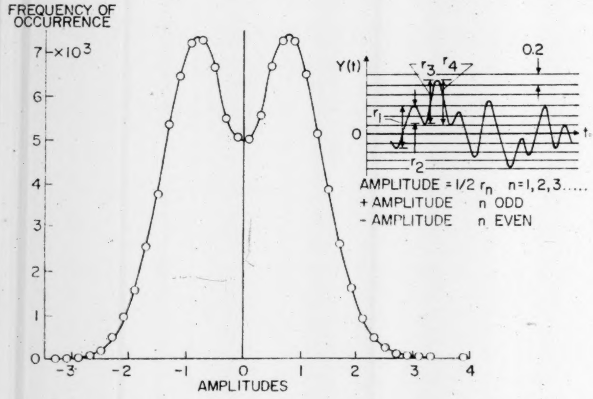

FIG. 4.—Distribution of Positive and Negative Amplitudes Regardless of Mean.
Band-limited white noise consisting of 320,000 random numbers.

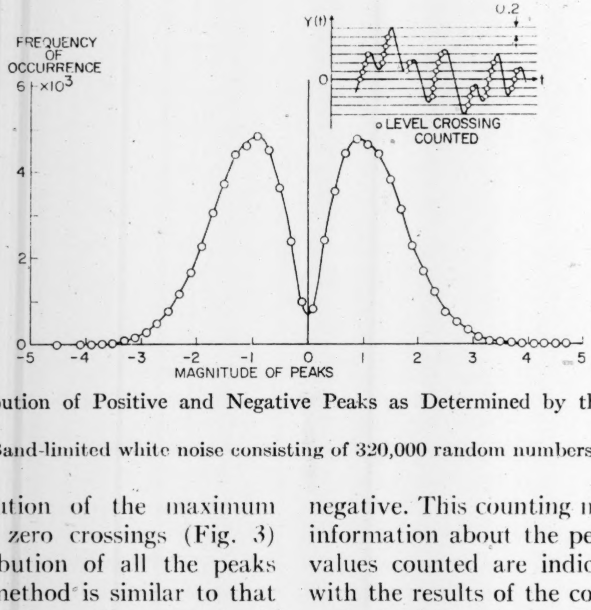

FIG. 5.—Distribution of Positive and Negative Peaks as Determined by the Level Crossing Method.

Band-limited white noise consisting of 320,000 random numbers.

Level Crossings.—This counting method and the following method afford other means of obtaining peak distributions which are most frequently used to program fatigue tests. This method records the number of times the time history crosses a given level with positive slope. It is logical to expect that the reference or zero axis of the time history will be crossed most frequently, with progressively fewer crossings occurring at the higher or lower levels. The number of peaks occurring in the interval between two adjacent levels (not necessarily the true number of peaks in the interval) was obtained by subtracting the respective number of crossings at each of these levels. Figure 5 shows the distribution of peaks obtained in this manner. It should be noted that there are relatively few peaks in the intervals close to zero. This can be explained by the fact that positive and negative peaks in the same interval cancel each other, leaving only positive or negative peaks in the interval (see Appendix II[4]). The same result can be obtained by subtracting, point by point, the two curves in Fig. 2. The curve for negative peaks reduces the curve for positive peaks significantly in the intervals near zero and vice versa.

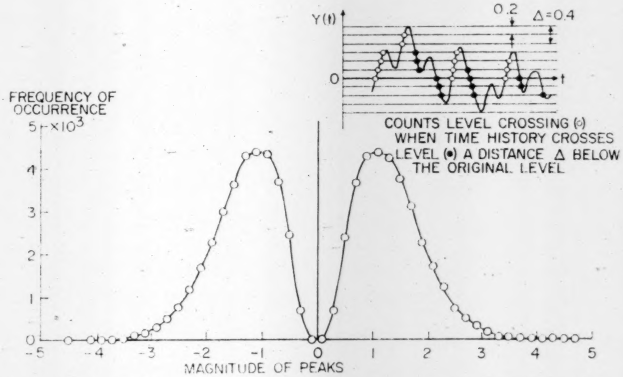

FIG. 6.—Distribution of Positive and Negative Peaks as Determined by the Level Crossings Eliminating Small Fluctuations Method.

Band-limited white noise consisting of 320,000 random numbers.

Level Crossings Eliminating Small Fluctuations.—This counting procedure is similar to the level crossing count method but must satisfy a secondary condition. Level crossings (positive slope) are counted only if the trace of the time history crosses some lower preset value a distance Δ below the level to be counted. In counting level crossings below the reference or zero axis (negative slope) the procedure is reversed; that is, level crossings are counted only if the trace crosses some preset value a distance Δ above the level to be counted. Level crossing of the zero axis can be counted using either a positive or negative slope. For this particular investigation a value of $\Delta = 0.4$ was selected. This increment corresponds to a stress interval in the testing program which is below the fatigue limit and thus insignificant with respect to fatigue. In essence, the preassigned value of Δ eliminates from the counting results all fluctuations in the time history having ranges (peak-to-peak) less than the value Δ. The results of the counting are presented in Fig. 6. As for level crossings, the peaks (again not necessarily the true number of peaks) were obtained by subtracting the number of crossings at adjacent levels. A comparison of the distribution of peaks obtained in this manner with that of the distribution of peaks obtained by level crossings will show the number of fluctuations with ranges less than 0.4 that have been eliminated. It should be remembered that occurrences in an interval are plotted at the midpoint of the interval.

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[4] See p. 732.

Means and Amplitudes.—This method of counting provides data that are generally regarded as most significant in fatigue. A mean is defined as one-half the algebraic sum of two adjacent peak values and an amplitude as one-half the algebraic difference between the same two adjacent peak values. This method provides a count of all the amplitudes about a corresponding mean value over the entire range of mean values. The counting method is illustrated in Fig. 7; three typical distributions of amplitudes and their corresponding means are presented. It should be noted that the distributions retain essentially the same shape regardless of the mean value selected. It should also be pointed out that this counting method can be used to obtain the results of some of the other counting methods—that is, peaks and amplitudes regardless of mean.

Means and Amplitudes Eliminating Small Fluctuations.—This counting method is similar to the mean-amplitude

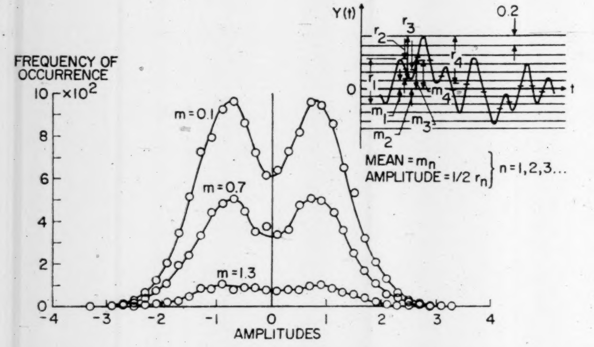


FIG. 7.—Distributions of Positive and Negative Amplitudes About a Specified Mean.
Band-limited white noise consisting of 320,000 random numbers.

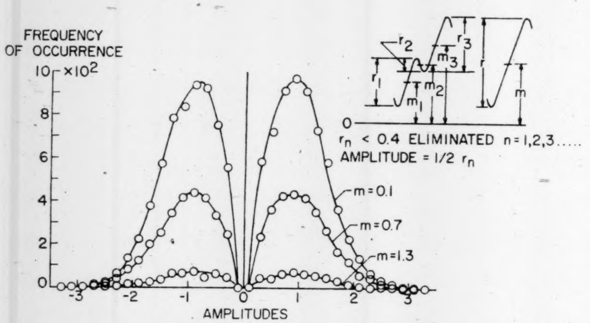


FIG. 8.—Distributions of Positive and Negative Amplitudes About a Specified Mean (Ranges <0.4 Eliminated).
Band-limited white noise consisting of 320,000 random numbers.

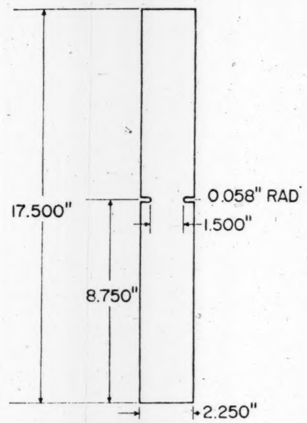

0.058" RAD
17.500"
1.500"
8.750"
2.250"

FIG. 9.—Sheet Specimen Details.

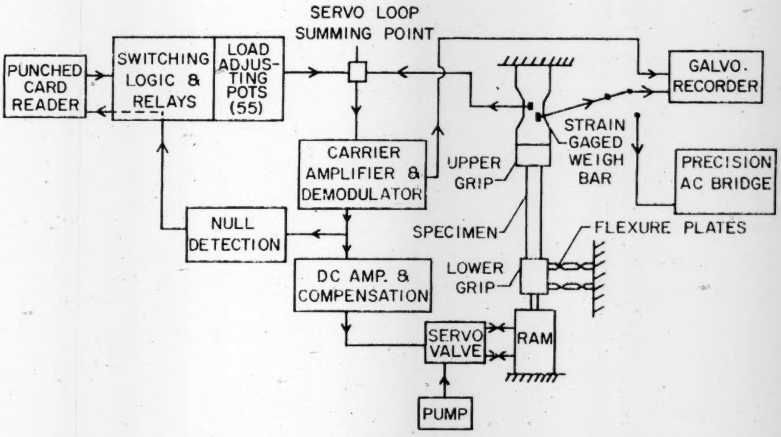

SERVO LOOP SUMMING POINT
PUNCHED CARD READER
SWITCHING LOGIC & RELAYS
LOAD ADJUSTING POTS (55)
GALVO. RECORDER
STRAIN GAGED WEIGH BAR
CARRIER AMPLIFIER & DEMODULATOR
UPPER GRIP
PRECISION AC BRIDGE
NULL DETECTION
SPECIMEN
FLEXURE PLATES
DC AMP. & COMPENSATION
LOWER GRIP
SERVO VALVE
RAM
PUMP

FIG. 10.—Block Diagram of Variable-Amplitude Testing Machine.

count method in that it counts the mean and amplitude between adjacent peak values. However, it was decided to eliminate all fluctuations having ranges less than 0.4 for the same reasons as discussed previously. At places in the time history where these small fluctuations occur (range < 0.4) a mean and amplitude between the peaks immediately before and immediately after these small fluctuations are obtained. Means and amplitudes for fluctuations with ranges greater than 0.4 are counted in the usual manner. The count is made as shown in Fig. 8, and three typical distributions of amplitudes and their corresponding mean values are presented. A comparison with Fig. 7 will show the number of fluctuations less than 0.4 that have been eliminated.

In the following paragraphs the statistics obtained by the above counting methods will be used to develop programmed load fatigue tests which simulate, with various degrees of complexity, the completely random-load fatigue test. It should be noted that an attempt has been made to determine some of the above statistics from the power spectrum of a random time history (1).

TEST PROCEDURES

Test Specimens:

Sheet specimens of 2024-T3 aluminum alloy were used for this investigation. The specimens contained edge notches which produced a theoretical elastic stress concentration $K_t = 4$. Figure 9 shows the specimen details. The material for these specimens is part of a stock of nominal 0.090-in. thick 2024-3T aluminum-alloy sheet retained at the Langley Research Center for fatigue test purposes.

Testing Machine:

A servohydraulic machine was developed for use in this investigation. A block diagram of the machine is shown in Fig. 10. The loading frame has a nominal capacity of ±20,000 lb in axial load. Cycling rates up to 7 cps are obtainable, depending on the load range. The important features of this programmed load fatigue machine are: (1) any type of load history defined by discrete load levels can be programmed in any arbitrary sequence using punched IBM cards; (2) 55 individually adjustable input channels, each identified by its own code, can be preset to any value between zero and full scale; and (3) a high degree of load accuracy can be obtained throughout the test.

In operation, the card reader transmits coded load information to the switching logic. Upon receipt of this information, the switching logic performs selected functional checks, then switches the desired input channel (preset 10-turn potentiometer) into the servoloop summing point. At the summing point, the voltage from the input channel is combined with the output from a load-sensing strain-gage bridge mounted on the weigh-bar which is in series with the specimen. The combined voltage from the summing point is fed into the carrier amplifier where the incoming voltage is compared with a reference voltage to determine the magnitude and polarity of the signal sent to the d-c amplifier. In the following discussion the signal sent to the d-c amplifier shall be called error which has both magnitude and polarity. The d-c amplifier uses the error to cause the servo valve to direct oil to one side of the ram, thus loading the specimen and thereby changing the output from the strain-gage bridge. The hydraulic oil flow is proportioned by the magnitude of error signal; thus as the load applied to the specimen approaches the desired load (error approaches zero), the flow is slowed so that the load approaches the desired load asymptotically. The minimum acceptable error is controlled by the settings of the load comparator. When the load error has been reduced to a value equal to or less than the comparator settings, the load comparator generates a signal which commands the reader to transmit the next piece of load information to the switching logic.

A circuit is provided which checks continuously for malfunction in any portion of the loading system. Specimen failure (complete separation of the specimen), detection of an error in the switching logic functional checks, or loss of command signal to the servovalve cause an immediate stop in oil flow by deenergizing the solenoid-controlled flow valve.

Loads are monitored by either a strip chart recorder or a null indicating a-c bridge. The strip chart is used to scan for extraneous loads, whereas the a-c bridge is used for static load measurements and to check system damping. The whole system is calibrated periodically, and static load indication is repeatable to 0.1 per cent of full scale. True load accuracy is thought to be ±0.2 per cent of full scale.

Three of these programmed load machines were used in this investigation.

Load Schedules:

Load schedules were developed using the statistical results of each of the previously described counting methods. In developing these load schedules, the following assumptions were made: (1) mean stress = 17.4 ksi (this corresponds to zero on the arbitrary scale used to "count" the time history), and (2) maximum stress = 43.5 ksi (this corresponds to +5.0 on the arbitrary scale). A description of the load schedules developed for each of three general types of tests follows:

Random Tests.—For load programs using a random distribution of peaks, the loads were programmed using 50 levels. These levels correspond to the 50 levels in the arbitrary scale for counting the time history. Three load programs were developed with a random distribution of peaks: (1) original generated time history, (2) maximum peaks between zero crossings, and (3) means and amplitudes eliminating small fluctuations ($\Delta = 0.4$). For these three load programs the sequence of load levels was preserved from the original generated time history. The fatigue life obtained in tests using the original generated time history will be used as the basis for determining whether or not a given simplification to the original time history retained the essential fatigue-inducing characteristics of the generated time history.

Eight-Step Constant Mean Block Tests.—Five load programs were developed with eight load steps and a constant mean. Because the distributions of negative and positive peaks were found to be approximately equal, only the positive distributions were used to develop load schedules. In all of the tests with eight steps and a constant mean each positive load was followed by an equal negative load. For these load programs the range of peaks between zero and maximum was divided into eight equal bands. Each band was then represented by a discrete load level at the middle of the band. The number of times each discrete load level was applied is defined by the intercepts of the band limits. One time through the distribution is defined as a "block" with the block being approximately 4000 load cycles for each counting method. Within a block, all of the cycles of load at each load level were applied consecutively with the order of load levels being varied randomly according to a schedule taken from a table of random numbers. The sequence of load levels was different for each block until the twentieth block; thereafter the schedule for the first 20 blocks was repeated. The sequence of load levels was the same for all tests. Eight-step constant mean block tests were conducted using the results of the following counting methods: (1) maximum peaks between zero crossings, (2) amplitudes regardless of mean, (3) level crossings, (4) level crossings eliminating small fluctuations, and (5) peak count (positive peaks > 0).

In (4) above, a value of $\Delta = 0.4$ was selected because this would eliminate stress cycles below the fatigue limit of the specimen. This is a reasonable approach because no damage is expected from stress cycles that have a maximum value less than the fatigue limit (6).

Eight-Step Variable Mean Block Tests.—The results of the amplitudes and means, and amplitudes and means eliminating small fluctuations, counting methods are obtained as distributions of amplitudes about various means. In order to develop load programs based on these statistics, the distributions of amplitudes for several means were grouped together into a combined distribution of amplitudes, about a fixed mean. For example, the distribution of amplitudes with means of -0.3, -0.1, $+0.1$, and $+0.3$ were combined into a distribution of amplitudes with a 0 mean. Similarly, distributions were obtained for three positive means and three negative means. The distribution for 0 mean was divided into eight equal amplitude bands in both the positive and negative direction. The representative amplitude for each band was selected in the middle of the band.

This then defines the values of 16 load setting potentiometers. To facilitate testing procedures, the remaining combinations of means and amplitudes were selected so that they could be applied by the proper sequencing of these 16 settings. Figure 11 shows schematically how this was accomplished.

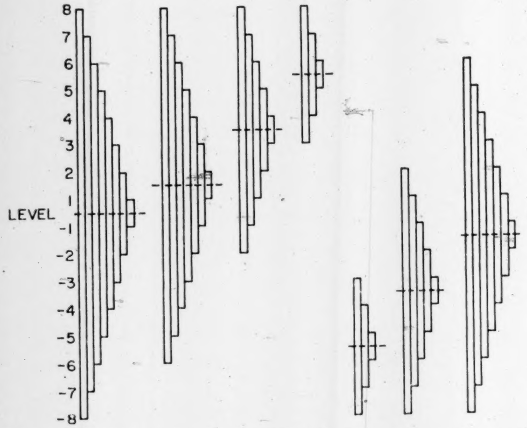


FIG. 11.—Schematic of Eight-Step Variable Mean Block Test.

As in the case of the constant mean block tests, each positive amplitude was followed by an equal negative amplitude. Also, the total number of cycles was approximately 4000 per block and the sequence of load combinations within a block was random.

Tests were conducted in this manner using the statistics obtained from the following counting methods: (1) means and amplitudes, and (2) means and amplitudes eliminating small fluctuations.

DISCUSSION OF RESULTS

The results of 60 variable-amplitude axial-load fatigue tests conducted on

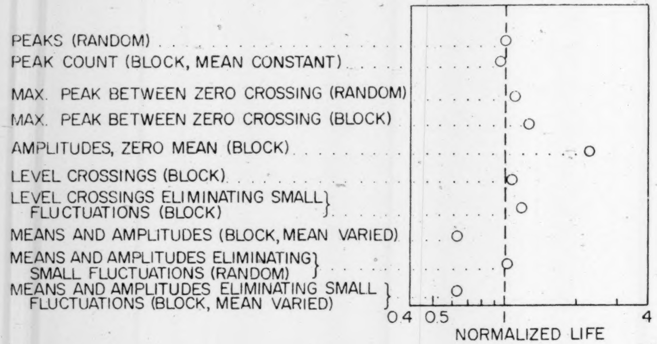


FIG. 12.—Normalized Results of Variable-Amplitude Fatigue Tests.

sheet specimens of 2024-T3 aluminum alloy are presented in Fig. 12. In Fig. 12 the symbols represent the geometric mean of six tests conducted using the same load program. The scatter in a given set of tests never exceeded $1\frac{1}{2}$ to 1 and was generally less than $1\frac{1}{3}$ to 1. This trend is in agreement with other variable-amplitude tests conducted at the NASA Langley Research Center (**6–8**).

Basis for Comparison:

The fatigue life obtained in random tests employing the original generated time history is assumed to be the life that would have been obtained in service. This life is compared with the life obtained by using the results of a simplifying counting procedure. In this way an evaluation can be made of the ability of the counting procedures to preserve the fatigue-inducing characteristics of the generated time history.

The results of all the tests were normalized on the results of tests conducted using the original generated time history (random). This was done by dividing the original time history into equal segments, each segment containing 5000 random numbers. The average number of peaks per 5000 numbers, for each of the different counting methods employed, was determined. The equivalent number of segments of the original time history required for failure was determined by dividing the total number of peaks required for failure for a given counting method by the average number of peaks per 5000 numbers as determined for the same counting method. The number of equivalent segments obtained in this manner, for each of the counting methods employed, was compared to the number of segments required for failure using the original time history.

The results of the variable-amplitude tests are summarized in Table 1.

Examination of the results obtained in this investigation leads to the general

TABLE 1.—RESULTS OF VARIABLE-AMPLITUDE TESTS.

Test Condition	Normalized Life	Remarks
Random:		
Peak count method	1.0	Generated time history
Maximum peak between zero crossings	1.10	See Note 1
Means and amplitudes eliminating small fluctuations	1.01	See Note 2
Eight-step, constant mean, block tests:		
Peak count method	0.97	See Note 3
Maximum peak between zero crossings	1.26	See Note 1
Amplitudes regardless of mean	2.28	See Note 4
Level crossings	1.07	See Note 2
Level crossings eliminating small fluctuations	1.18	See Note 2
Eight-step, variable mean, block tests:		
Means and amplitudes	0.63	See Note 5
Means and amplitudes eliminating small fluctuations	0.63	See Note 5

NOTE 1.—The maximum peaks between zero crossings method eliminates approximately 25 per cent of the peaks in a given segment of generated time history. The magnitude of these peaks can be reasonably large and their elimination would be expected to increase fatigue life. The difference between results obtained in the random tests and the eight-step constant mean, block tests is probably due to an order effect in the block tests. Tests based on this counting method appear to lead to longer lives than the random time histories they are to represent.

NOTE 2.—These counting methods, in general, eliminate small fluctuations near the zero mean, which have been shown to have a very small effect on fatigue life (6).

NOTE 3.—A shorter normalized life was anticipated. In these tests, positive peaks were treated as though each occurred between zero crossings, thus making this type of test more damaging than the generated time history.

NOTE 4.—When the amplitude is retained but the mean discarded, the damaging effect of mean stress variations is reduced and therefore a longer fatigue life results. This method of service load simulation may lead to grossly unconservative answers.

NOTE 5.—This result was very surprising. The reason for this short life is not obvious although the deleterious effect of the mean stress probably is an important factor.

conclusion that the majority of the counting procedures employed do successfully retain the essential fatigue characteristics of the particular time history investigated. However, it is important to note that the answers obtained in this investigation were for one type of load history and one series of tests for which certain assumptions were made. It is probable that different answers would have been obtained if (1) the generated time history had vastly different statistical properties than the one used, (2) a different range of stress values had been used, or (3) different assumptions had been made for such variables as block size, location of the representative amplitude within the amplitude band, and number of stress levels to represent a given distribution. The effects of some of these variables on fatigue life are discussed in references (6), (7), and (8).

SUMMARY

A method has been presented for evaluating the ability of several counting procedures to retain the essential fatigue-inducing characteristics of a given random time history. A digitally generated random time history having an arbitrarily shaped power spectrum (in this case band-limited white noise) was analyzed using several different counting methods, and the results obtained were used to program axial-load fatigue tests on notched sheet specimens.

The results of the fatigue tests were normalized on the life obtained in tests using the generated time history (random), and ranged from 0.63 for block tests using 38 combinations of means and amplitudes to 2.28 for block tests in which all amplitudes were applied about a common mean. Although correlation was very good for some cases, many additional tests are needed on time histories having different power spectra before general conclusions can be reached as to the ability of a given counting method to retain the fatigue-inducing characteristics of a time history.

APPENDIX I

DETERMINATION OF COEFFICIENTS OF A FOURIER COSINE SERIES REPRESENTATION OF FREQUENCY-RESPONSE FUNCTION

Consider the continuous periodic function of time, of amplitude A and frequency f, such that

$$y(t) = A \cos 2\pi f t$$

If this is sampled at discrete time intervals Δt, the continuous time history is replaced by a discrete set of values y_i equal to $y(t)$ when $t = i\Delta t$ and undefined in between. Thus

$$y(t) = y_i = A \cos 2\pi f i \Delta t \quad \text{for } t = i\Delta t$$

$$y_i = A \cos 2\pi f i \Delta t \frac{f_F}{f_F}$$

$$\text{where } f_F = \frac{1}{2\Delta t}$$

$$y_i = A \cos 2\pi \frac{f}{f_F} \frac{i\Delta t}{2\Delta t}$$

$$y_i = A \cos i\pi \frac{f}{f_F} \qquad \text{(3)}$$

The above time history can be modified to change its frequency characteristics (that is, numerically filtered) as follows:

$$Y_i = \sum_{K=-M}^{M} a_K y_{i+K} \qquad \text{(4)}$$

where:

Y_i = filtered time history,

y_{i+K} = original time history, and

a_K = filter factors or coefficients.

Equation 4 represents, in numerical form, the passage of an input signal, $y(t)$, through some linear system which results in an output signal $Y(t)$ (**4**).

Upon substituting y_i (Eq 3) for y_{i+K} (Eq 4),

$$Y_i = A\Bigg[a_{-M}\cos\pi\frac{f}{f_F}(i-M)\cdots$$

$$+ a_{-K}\cos\pi\frac{f}{f_F}(i-K)\cdots$$

$$+ a_0\cos\pi\frac{f}{f_F}\,i\cdots$$

$$+ a_K\cos\pi\frac{f}{f_F}(i+K)\cdots$$

$$+ a_M\cos\pi\frac{f}{f_F}(i+M)\Bigg]$$

$$Y_i = A\Bigg\{a_0\cos\pi\frac{f}{f_F}\,i$$

$$+ \sum_{K=1}^{M}\Bigg[a_{-k}\cos\pi\frac{f}{f_F}(i-K)$$

$$+ a_K\cos\pi\frac{f}{f_F}(i+K)\Bigg]\Bigg\}$$

$$Y_i = A\cos\pi\frac{f}{f_F}\,i\left(a_0 + 2\sum_{K=1}^{M}a_K\cos\pi\frac{f}{f_F}K\right)$$

$$Y_i = y_i\left(a_0 + 2\sum_{K=1}^{M}a_K\cos\pi\frac{f}{f_F}K\right)\ldots\ (5)$$

The term in parentheses is in the form of a Fourier cosine series. It is therefore necessary to represent the frequency response function $F(f/f_F)$ in the form of a Fourier cosine series in order to filter the generated time history. This was done as follows: let

$$\frac{f}{f_F} = \frac{h}{H} \quad \text{where} \quad h = 0, 1, 2, \cdots H$$

Then:

$$F\left(\frac{f}{f_F}\right) \to F\left(\frac{h}{H}\right) = F_h = \frac{A_0}{2}$$

$$+ \sum_{h=1}^{H} A_n\cos\frac{\pi nh}{H}$$

where:

$$A_n = \frac{2}{H}\int_0^H F_h\cos\frac{\pi nh}{H}\,dh$$

Using the trapezoidal rule of numerical integration

$$\{A_n\} = \frac{2}{H}\left[\cos\frac{\pi nh}{H}\right][I_T]\,\{F_h\}$$

where:

$$[I_T] = \begin{bmatrix} \frac{1}{2} & 0 & 0 & \cdots & & & \\ 0 & 1 & & & & & \\ 0 & & 1 & & & & \\ \vdots & & & \ddots & & & \\ \vdots & & & & \ddots & & \\ \vdots & & & & & 1 & \\ \vdots & & & & & & \frac{1}{2} \end{bmatrix}$$

Solving for $\{F_h\}$,

$$\{F_h\} = \frac{H}{2}\,[I_T]^{-1}\left[\cos\frac{\pi nh}{H}\right]^{-1}\{A_n\}$$

Define:

$$\left[\cos\frac{\pi nh}{H}\right]^{-1} = \frac{2}{H}\,[I_T]\left[\cos\frac{\pi nh}{H}\right][I_T]$$

then

$$\{F_h\} = \left[\cos\frac{\pi nh}{H}\right][I_T]\,\{A_n\}$$

Therefore

$$F_h = \frac{A_0}{2} + \sum_{h=1}^{H-1}A_n\cos\frac{n\pi h}{H} + \frac{A_H}{2}(-1)^h$$

Finally the coefficients A_n are related to the filter factors a_K as follows:

$$a_0 + 2\sum_{k=1}^{M}a_k\cos\pi\frac{f}{f_F}k = \frac{A_0}{2}$$

$$+ \sum_{h=1}^{H-1}A_n\cos\frac{\pi nh}{H} + \frac{A_H}{2}(-1)^h$$

and from equating coefficients

$$a_0 = \frac{A_0}{2},\quad a_1 = \frac{A_1}{2},\quad a_2 = \frac{A_2}{2},\quad \cdots,$$

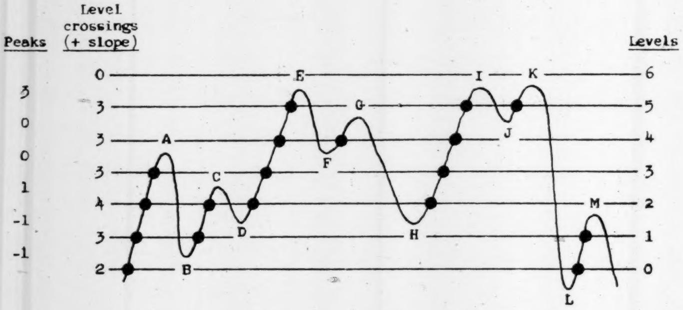
$$a_h = \frac{A_h}{2},\quad a_H = \frac{A_H}{4}$$

APPENDIX II

PEAKS OBTAINED FROM LEVEL CROSSING COUNT METHOD

As mentioned in the text, the level crossing count method is one of several methods used to obtain a peak count. This method does not, however, provide an accurate count of peak values, particularly near the mean or reference axis of the time history. The reason cited was that negative and positive peaks in a given interval tend to cancel each other, leaving only positive or negative peaks in the interval. The following sketch will illustrate that positive and negative peaks in a given interval do cancel each other, resulting in an erroneous peak count.

Points *A*, *C*, *E*, *G*, *I*, *K*, and *M* are considered to be positive peaks, while *B*, *D*, *F*, *H*, *J*, and *L* are considered to be negative peaks. Counting level crossings with positive slope, the results are as indicated to the left of the time history. Peaks are then obtained by subtracting the number of level crossings at adjacent levels and are indicated at the left of the time history. It should be remembered that, of the two adjacent levels the higher level is subtracted from the lower level. This procedure will determine whether the peaks in the interval are positive or negative. When the peaks are visually counted in the intervals, the following values are obtained and are compared with the peak count from the level crossing method:



Interval	6–5		5–4		4–3		3–2		2–1		1–0	
Peaks	+	−	+	−	+	−	+	−	+	−	+	−
Visual	3	0	1	1	1	1	1	0	1	2	0	1
Level crossings	3	0	0	0	0	0	1	0	0	1	0	1

In intervals 5–4 and 4–3 a positive and a negative peak occur but are not counted by the level crossing method. In the interval 2–1 two negative peaks and one positive peak occur but the level crossing method only counts one negative peak for that interval. Thus it appears that positive and negative peaks in a given interval cancel each other, leaving only positive or negative peaks in the interval. Therefore the level crossing method does not give a true indication of the peaks.

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DISCUSSION

MR. T. J. DOLAN.[1]—I should like to ask the authors how many specimens were used in the fatigue experiments to determine the "life," and how were the results expressed (that is, as mean values, median values, or some other statistic)? It would be helpful if the authors could clarify the method used in calculating the "normalized life" for comparing the results from the various methods of simulating the random history in their fatigue tests. Their development of equipment and realistic methods of simulating random loadings in service are important steps in the rational evaluation of methods for predicting cumulative fatigue damage and studying the history effects in the loading sequence.

MR. H. A. LEYBOLD (*author*).—The "life" for each test condition was determined by obtaining the geometric mean of the number of load cycles required for failure in each of six identical tests. The geometric mean was determined by taking the antilog of the mean of the logs of the cycles to failure. The lives obtained in this manner were then compared with the life obtained when using the original generated random time history.

In order to make the comparison, an "equivalent life" was obtained by first dividing the original time history into equal segments, each segment containing 5000 random numbers. Then the average number of load cycles (or one-half the number of peaks) per 5000 number was determined for each of the different counting methods employed. From the test results, the total number of load cycles required for failure could be determined for each particular counting method. From this information the equivalent number of segments of the original random time history required for failure could be determined. The number

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[1] Professor and Head, Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

of equivalent segments obtained in this manner, for each of the counting methods employed, was then compared with the number of segments required for failure using the original time history.

MR. E. J. GRIFFIN.[2]—Does the author have any comments on the differences in fatigue life that would be obtained from stationary random processes with the same spectral density and values of root mean squared intensity—that is, processes that are different in the time domain but the same in the frequency domain?

MR. LEYBOLD.—A study of the effects on fatigue life of changing the time domain while keeping the frequency domain a constant has not been made, thus far. Some work in this area is anticipated in the future. However, the position of infrequent high loads in the time domain may be expected to have a systematic effect on fatigue life. For example, if high loads are encountered very early in the time history the fatigue life will probably increase due to the beneficial residual stresses produced at notches. In contrast, if high loads are encountered shortly after the initiation of a fatigue crack, failure may occur if the residual static strength of the specimens is exceeded. Systematic effects on fatigue life of other parameters in the time domain remain to be investigated.

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[2] **Chief, Structures Section, Flight Standards Service, Federal Aviation Agency, Washington, D. C.**

THERMAL FATIGUE—PART I. AN ANALYSIS OF THE CONVENTIONAL EXPERIMENTAL METHOD

By A. E. Carden[1]

Synopsis

An analysis of the conventional (Coffin type) thermal fatigue test is presented by describing an analytical model from which three methods for determining the plastic strain range are derived. One method for calculating plastic strain does not require the knowledge or use of the thermal coefficient, modulus of elasticity, or stress range. The accuracy is not affected by the variation of the thermal coefficient, modulus of elasticity, or yield strength during the heating and cooling periods. Plastic strains resulting from yielding and relaxation may be accounted for by this method. The errors of conventional methods are discussed. A method for plotting the stress-strain hysteresis loop is also given.

From an analysis of the load-temperature loop, it is clearly shown how all significant variables can be determined without plotting a stress-strain hysteresis loop. The plastic strain range, the mechanical strain range (elastic plus plastic), the stress range, and the plastic strain energy per cycle can easily be determined from the load-temperature loop.

Nomenclature

A, cross-sectional area, sq in.
C, K, n, constants
D, dial gage reading, in.
E, modulus of elasticity, psi
F, degree of constraint factor
L, length, in.
N, cycle number
P, load, lb
R, radius, in.
W, energy, in-lb or in-lb per cu in.
ROA, reduction of area, per cent
T, temperature, deg F
t, time, sec
δ, total strain, in.
ϵ, unit strain, dimensionless
σ, unit stress, psi
α, coefficient of thermal expansion, per deg F

Subscripts:

g, geometric strain, lineal displacement
m, mechanical strain, sum of elastic and plastic components
e, elastic strain, recoverable

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[1] Assistant Professor, Department of Engineering Mechanics, University of Alabama, University, Ala

p, plastic strain, nonrecoverable
T, thermal strain
s, spring representing restraint system
f, designation of failure
0, designation of the value at zero load
x, r, z, coordinate directions
t, tensile value
c, compressive value

Superscripts:
deg, °, designates value for unrestrained cycling
prime, ′, designates value for restrained cycling

The volume of a solid body increases or expands when the temperature of the body is raised. This change of volume is called "thermal expansion," and the term "thermal strain" refers to the unit change of a lineal dimension associated with the rise in temperature. No internal stress is produced in a solid material if the temperature increase is the same in all parts of the body and if the volume change is unrestrained.

In a solid that is restrained so that it cannot expand (or contract) freely, a temperature change gives rise to a stress called a "thermal stress." A mechanical deformation is uniquely related to and concurrent with a thermal stress. A thermal stress deformation that is recovered or relieved when the restraint is removed is said to be an elastic deformation, while one that is not recovered when the restraint is removed is said to be a plastic deformation. Plastic deformations can result from large thermal stresses, high temperatures, or long duration of stress application.

The elastic and plastic deformation together constitute the internal, or mechanical load, strain which will hereafter be referred to simply as mechanical strain. The mechanical strain at a point is always related to the stress at that point and this stress can be produced independent of temperature. A mechanical strain is not uniquely dependent on temperature, but a thermal strain is. A thermal strain can occur without producing a thermal stress (that is, in an unrestrained body), but a thermally induced stress (in a restrained body) always produces (or is accompanied by) a mechanical strain.

In thermal fatigue tests there are therefore three kinds of strain: thermal strain, mechanical strain, and geometric strain. A thermal strain is that strain produced by temperature change. A mechanical strain is that strain which is produced by (or is concurrent with) a stress, and there is no intrinsic difference between a thermal stress and a mechanical stress. A geometric strain is that which can be physically measured by a displacement. The geometric, or observed, strain is the algebraic sum of the mechanical strain and the thermal strain.

A repeated fluctuation of temperature in a restrained solid produces cyclic thermal stresses (and therefore cyclic mechanical strains), leading ultimately to failure. The name "thermal fatigue" is applied to failures produced by cyclic thermal stresses. The cyclic mechanical strains associated with the thermal stresses may be only elastic or they may contain both elastic and plastic components. The significant variables for thermal fatigue are stress, time, mechanical strain (or frequently the plastic strain component), maximum temperature, and temperature range.

The conventional thermal fatigue

experiment can be simply described as the alternate heating and cooling of a thin-walled tubular specimen in a device that restricts the longitudinal movement of the specimen.

A timely and significant critical review of thermal fatigue by Yen (**1**)[2] is primarily an appraisal of some of the significant results of thermal fatigue experiments. The article includes a brief discussion of the test method and summarizes the two major difficulties of the conventional thermal fatigue test: (1) the lack of a uniform temperature throughout the length of a tubular specimen; and (2) the determination of the mechanical strain, especially the plastic component, produced by restrained thermal cycling. This paper is primarily concerned with the second of these difficulties.

For several years thermal fatigue testing was pursued with vigor and gained widespread attention. Interest abated, however, with unsuccessful efforts to obtain good agreement between the experimental results and the lives of service applications, or between thermal fatigue and isothermal strain fatigue data. No satisfactory explanation of the dilemma was universally accepted, and a shadow of doubt began to cloud the experimental method.

The value of the thermal fatigue test has been contested because many believe that the published methods of determining the plastic strain values are questionable; that any calculated value of plastic strain may be gravely in error; that the construction of a stress-strain diagram is not possible; and that the

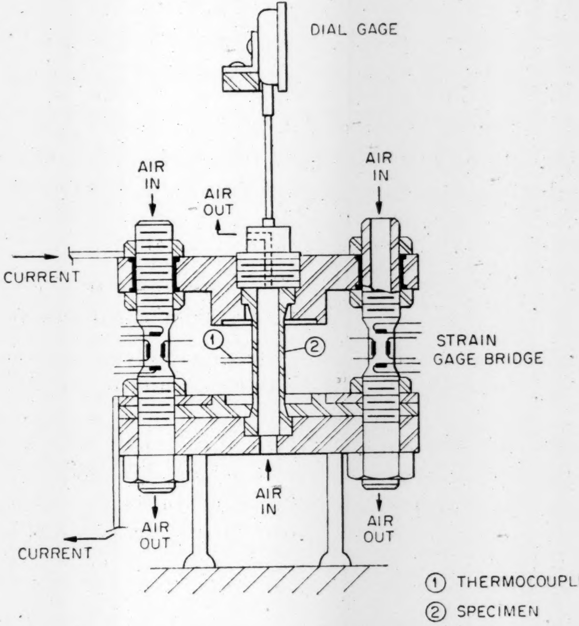

FIG. 1.—Schematic Diagram of the Conventional Thermal Fatigue Testing Machine.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

plastic strain per cycle indeed becomes an illusive quantity to calculate. The reasons for doubting the validity of the results of the conventional test have not rested simply on prejudice. The thermal coefficient, modulus of elasticity, and strength properties all vary with temperature, which in this test varies with time. Also, at any instant of time the temperature varies throughout the length of the specimen. Thus the problem has appeared to be too complex to deduce the stress-strain cycle of the material.

Thermal fatigue reports often do not relate how the plastic strain values were calculated and are vague about how other data were obtained. A clear, critical analysis of the published methods of determining the plastic strain has not yet appeared. The possible reasons why thermal fatigue data do not always show good agreement with isothermal strain fatigue results are generally not discussed. A need exists to extend Yen's review to analyze the experimental problem and to suggest possible alternatives.

The purpose of this paper is to present a simplified model of the conventional thermal fatigue test and, using this model, to develop a method for determining the stress-strain cycle and to deduce several techniques for calculating the magnitude of the plastic strain.

THERMAL FATIGUE TESTS

In the conventional thermal fatigue test a thin-walled tubular specimen is alternately heated and cooled in an apparatus that restricts the longitudinal movement of the specimen. A typical testing machine is shown in Fig. 1. The straight portion of the specimen length is commonly 2 in. and at each end of the gage length are two fillets that constitute about one third of the total length. Usually a large electric current passing through the specimen is the source of heat and a stream of compressed gas passing through the central hole cools the specimen. The axial load is determined from two load cells that restrain the thermal movement of the specimen. Because the restraint system is not perfectly rigid, a dial gage is used to measure the small displacement of the specimen ends during restrained cycling. Due to the heat sink at each end of the specimen, the temperature throughout the length is never uniform. Because direct gaging of the mechanical strain during thermal cycling is not possible, the determination of the stress-strain cycle becomes one of deduction. The temperature of the midlength of the specimen, the axial load, and the displacement of the ends of the specimen are generally the only measurements available.

ANALYSIS OF THE THERMAL FATIGUE TEST: SIMPLIFIED MODEL

The methods to be presented for calculating the value of the plastic strain range are based on a simplified model of the typical thermal fatigue test. The nomenclature for this model is defined at the beginning of the paper.[3] The necessity for each of the details of this model will be discussed when the model is compared to the actual case.

General:

The reader should study the details of the graphic representation of this model shown in Figs. 2 and 3. The specimen consists of two segments, each having uniform but unequal cross-sectional areas designated by appropriate subscripts. This specimen is uniformly cycled between the temperatures T_a and T_d, and has an elastoplastic mechanical behavior. The thermal coefficient of expansion is constant. The specimen is anchored to a rigid end at section *0* and can be attached at section *2* to a spring having a constant, K_s. With the spring removed, the geo-

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[3] See p. 735.

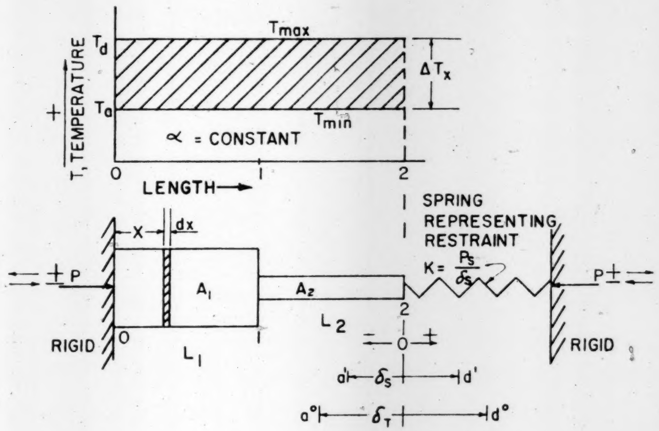

T, TEMPERATURE
+
T_d
T_a
T_max
T_min
ΔT_x
∝ = CONSTANT
0
1
2
LENGTH→
SPRING REPRESENTING RESTRAINT
K = P_s/δ_s
X
dx
±P
P±
A_1
A_2
L_1
L_2
RIGID
RIGID
0
1
2
0
d'
δ_s
d'
a°
δ_T
d°

FIG. 2.—Model of the Conventional Thermal Fatigue Test.

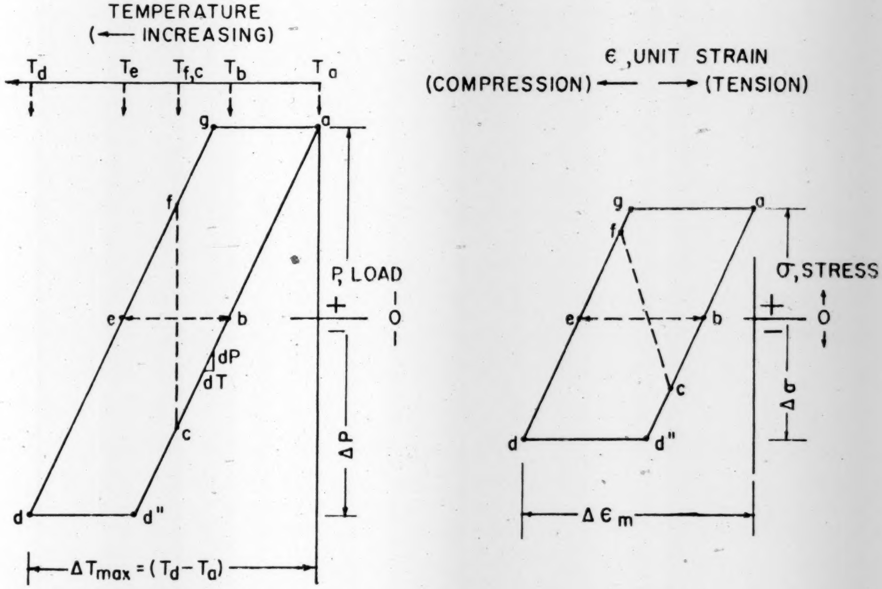

TEMPERATURE
(← INCREASING)
T_d
T_e
T_f,c
T_b
T_a
g
a
f
e
b
dP
dT
c
d
d"
P, LOAD
+
0
−
ΔP
ΔT_max = (T_d − T_a)
ε, UNIT STRAIN
(COMPRESSION) ← → (TENSION)
g
a
f
e
b
c
d
d"
σ, STRESS
+
0
−
Δσ
Δε_m

FIG. 3.—Hysteresis Loops for Area A_2 of the Thermal Fatigue Model.

metric displacement of point *2* during the temperature rise from *a* to *d* is:

$$\delta_T = \int_0^2 \int_a^d \alpha(dT)\, dx \ldots\ldots (1)$$

The displacement, δ_T, is equal to the area between temperature profiles multiplied by α. Since dT is a constant over the length *0–2*, and α is constant, the total thermal strain is

$$\delta_T = \alpha(T_d - T_a)\,(L_1 + L_2) \ldots\ldots (2)$$

The specimen is heated to an intermediate temperature between T_a and

T_d, and at this temperature the spring is attached such that zero load exists in the specimen. A geometric displacement of point *2* is observed as the temperature cycles. This displacement is also the deflection δ_s of the spring, and:

$$\delta_s = \frac{P_d - P_a}{K_s} \qquad (3)$$

The difference of these two displacements δ_T and δ_s is therefore the total mechanical strain sustained by the specimen as a result of the change in load, $P_a - P_d$. This mechanical strain δ_m produced by the loading effect of the restraint is the sum of the elastic and plastic components:

$$\delta_m = \int_0^2 \epsilon_e \, dx + \int_1^2 \epsilon_p \, dx \qquad (4)$$

It is clear that the displacement of point *2* is also the geometric strain of the reference length $(L_1 + L_2)$. The geometric strain is the sum of the mechanical and thermal strain, or

$$-\delta_s = \delta_m + \delta_T \qquad (5)$$

The sign convention adopted here is this: thermal and mechanical strains with positive signs are associated with positive temperature changes and tensile strains in the specimen respectively. Therefore a positive geometric displacement of point *2* is associated with a decreasing load in the spring. A tensile load in the model specimen will always occur with a tensile load in the elastic element of the model. In the model, therefore, the geometric strain term δ_s must be preceded with a negative sign as shown in Eq 5. In the conventional test, however, the elastic element is in parallel with the specimen rather than in series and the sign of the δ_s term must be changed accordingly.

Because the temperature is uniform at any time, *t*, a plot of temperature versus load will produce a loop resembling that shown in Fig. 3. The total mechanical strain during an increment of change in temperature T_a to T_b is:

$$-\delta_m]_a^b = \delta_T]_a^b + \delta_s]_a^b \qquad (6)$$

$$-\delta_m]_a^b = \alpha(T_b - T_a)\,(L_1 + L_2) + \frac{P_b - P_a}{K_s} \qquad (7)$$

From the *P-T* loop any one of several methods can now be employed to determine the plastic-strain range, $\Delta\epsilon_p$, without plotting the stress-strain curve. The plastic strain range is equal to the maximum internal width of the stress-strain loop shown in Fig. 3, and is sometimes called the plastic strain per half cycle.

Method I.—The total plastic strain per half cycle, $\Delta\delta_p$, is equal to the residual mechanical strain after the excursion of heating and cooling for the half cycle (*b-c-d″-d-e*). This plastic strain is the strain that remains in the specimen after heating from zero load (point *b*) to $T_{\max}$ (point *d*) and cooling to zero load (point *e*). From Eq 7 the total plastic strain per half cycle is:

$$-\Delta\delta_p = \alpha(T_e - T_b)\,(L_1 + L_2) + \frac{P_e - P_b}{K_s} \qquad (8)$$

Since $P_b = P_e = 0$,

$$-\Delta\delta_p = \alpha(T_e - T_b)\,(L_1 + L_2) \qquad (9)$$

Because the length L_2 is reduced in cross-section and the material is elastoplastic, yielding will occur only in the length L_2. If the unit plastic is uniformly distributed the plastic strain range in the length L_2 is:

$$\Delta\epsilon_p = -\alpha(T_e - T_b)\,\frac{(L_1 + L_2)}{L_2} \qquad (10)$$

The significance of Eq 10 is that the width of the load-temperature loop at zero load is directly related to the plastic strain range. The value $(L_1 + L_2)$ is the total length that contributes to the thermal strain of the specimen, and L_2 is the only length that sustains plastic flow. That the length sustaining plastic flow is not equal to the length undergoing thermal expansion is noteworthy.

Method II.—The plastic strain range can also be computed by calculating the mechanical strain from the minimum temperature (point *a*) to the maximum temperature (point *d*) and subtracting the elastic strain component. The total mechanical strain from Eq 4 is the sum of the elastic and plastic components, or:

$$-(\delta_e + \delta_p) = \delta_T + \delta_S \ldots\ldots (11)$$

or

$$-\delta_p = \alpha(\Delta T)\,(L_1 + L_2) + \frac{\Delta P}{K_s} + \frac{\Delta P}{E}\left(\frac{L_1}{A_1} + \frac{L_2}{A_2}\right) \ldots\ldots (12)$$

Since $\delta_p = \epsilon_p L_2$, then

$$-\Delta\epsilon_p = \alpha(\Delta T)\,\frac{(L_1 + L_2)}{L_2} + \frac{\Delta P}{K_s L_2} + \frac{\Delta P}{E L_2}\left(\frac{L_1}{A_1} + \frac{L_2}{A_2}\right) \ldots\ldots (13)$$

If the ratio L_1/A_1 is much less than L_2/A_2, then

$$-\Delta\epsilon_p \approx \alpha(\Delta T)\,\frac{(L_1 + L_2)}{L_2} + \frac{\Delta P}{K_s L_2} + \frac{\Delta\sigma_2}{E} \ldots (14)$$

In Eqs 12 to 14 ΔT, ΔP, $\Delta\sigma_2$ are the range of values and equal to the maximum minus the minimum value. Positive changes of temperature are associated with decreasing values of stress and load. Equation 13 clearly shows the influence of the specimen geometry and of the elasticity of the restraint on the magnitude of $\Delta\epsilon_p$.

Method III.—If a constant temperature line through the loop of Fig. 3 intersects the elastic (linear) portion of both the heating and cooling regions (points *c* and *f*, respectively), then only the difference in load is required to calculate the plastic strain range. From Eq 5,

$$-\delta_m\Big]_c^f = \alpha(T_f - T_c)\,(L_1 + L_2) + \frac{P_f - P_c}{K_s} \ldots\ldots (15)$$

but $T_c = T_f$; therefore,

$$-\delta_m\Big]_c^f = \frac{P_f - P_c}{K_s} \ldots\ldots\ldots (16)$$

or

$$\delta_p\Big]_c^f = -\frac{P_f - P_c}{K_s} - \frac{P_f - P_c}{E}\left(\frac{L_1}{A_1} + \frac{L_2}{A_2}\right) \ldots (17)$$

or

$$\Delta\epsilon_p = -\frac{P_f - P_c}{L_2}\left(\frac{L_1}{A_1 E} + \frac{L_2}{A_2 E} + \frac{1}{K_s}\right) \ldots (18)$$

Again, if $L_1/A_1 \ll L_2/A_2$, then

$$\Delta\epsilon_p \approx -\frac{\Delta P}{K_s L_2}\Big]_c^f - \frac{\Delta\sigma_2}{E}\Big]_c^f \ldots\ldots (19)$$

The P-T Loop of the Model:

A slight digression may be of benefit at this point to extend the analysis of the load-temperature (*P-T*) loop in order to determine the slope of the *P-T* curve, dP/dT, at zero load. At zero load the increment of plastic strain is zero for a small increment of load and the mechanical strain is only elastic. From Eq 5 and

$$\Delta\delta_e = \frac{\Delta P}{E}\left(\frac{L_1}{A_1} + \frac{L_2}{A_2}\right)$$

an infinitesimal increment of $d(\delta m)$ accompanying an increment of temperature change, dT, can be written as

$$-dP\left(\frac{L_1}{A_1E}+\frac{L_2}{A_2E}\right) = \alpha(dT)\,(L_1+L_2)+\frac{dP}{K_s}\ldots\ldots\ldots(20)$$

or

$$-dP\left(\frac{L_1}{A_1E}+\frac{L_2}{A_2E}+\frac{1}{K_s}\right) = \alpha(dT)\,(L_1+L_2)\ldots\ldots(21)$$

and

$$\left.\frac{dP}{dT}\right|_{P=0} = \frac{-\alpha(L_1+L_2)}{\left(\frac{L_1}{A_1E}+\frac{L_2}{A_2E}+\frac{1}{K_s}\right)}\ldots(22)$$

The slope of the *P*-*T* loop at zero load is a function of α, E, K_s, and the lengths and areas of the segmented specimen.

If perfect rigidity were attainable, the term $1/K_s$ would be zero. By substitution of typical values into Eq 22, it can be shown that about one third of the slope of P versus T stems from the term $1/K_s$. Thus it is indicated that the deflection of the restraint system must be considered in the methods presented.

The fact that the constraint system has an appreciable deflection and that δ_m will always be less than δ_T does not imply that the mechanical strain range, $\Delta\epsilon_m$, is always less than $\alpha(\Delta T)$; indeed, $\Delta\epsilon_m$ is usually greater than $\alpha(\Delta T)$. From Eq 5 it follows that

$$-\epsilon_{m1}L_1-\epsilon_{m2}L_2 = \alpha(\Delta T)\,(L_1+L_2)+\frac{\Delta P}{K_s}\ldots\ldots(23)$$

and

$$-\Delta\epsilon_{m2}L_2 = \alpha(\Delta T)\,(L_1+L_2)+\frac{\Delta P}{K_s}+\frac{\Delta PL_1}{A_1E}\ldots\ldots(24)$$

or

$$-\Delta\epsilon_{m2} = \alpha(\Delta T)\,\frac{(L_1+L_2)}{L_2}+\frac{\Delta P}{L_2}\left(\frac{1}{K_s}+\frac{L_1}{A_1E}\right)\ldots(25)$$

In other words, the mechanical strain in length L_2 is greater than $\alpha(\Delta T)$, provided that the second term of Eq 25 does not nullify the effects of the factor $(L_1+L_2)/L_2$ which is always greater than one; that is, if

$$\Delta P\left(\frac{1}{K_s}+\frac{L_1}{A_1E}\right) < \alpha\Delta TL_1\ldots\ldots(26)$$

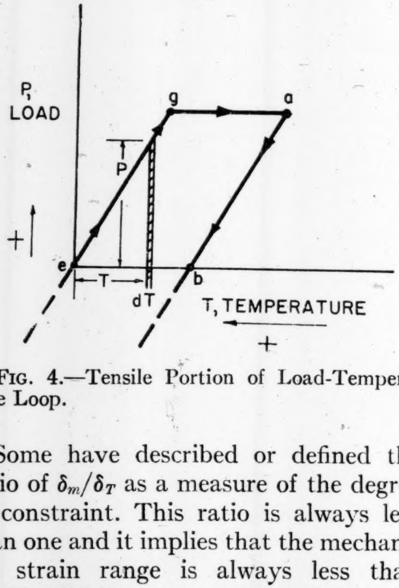


FIG. 4.—Tensile Portion of Load-Temperature Loop.

Some have described or defined the ratio of δ_m/δ_T as a measure of the degree of constraint. This ratio is always less than one and it implies that the mechanical strain range is always less than $\alpha(\Delta T)$. This is not true. It would be far better to define the degree of constraint by the ratio of $(\Delta\epsilon_{m2})/(\alpha(\Delta T))$. This ratio of the unit mechanical strain at the gage section during some excursion of temperature ΔT to the value of uninhibited thermal strain over the same temperature range is usually greater than one. The degree of constraint is represented by the factor F. The mechanical strain range is therefore equal to the uninhibited thermal strain $\alpha(\Delta T)_{\max}$ times the factor F. The constraint factor F is dependent on the lengths and areas of the specimen, α, E, and K_s.

Plastic Strain Energy from the P-T Loop.—The increment of total strain energy, dW, put into the system during the increment of temperature change dT of Fig. 4 is $P \cdot d(\delta_m)$. The mechanical strain may be obtained from Eq 5, and $dW = P \cdot d(\delta_T + \delta_s)$ or

$$dW = P\alpha(dT)(L_1 + L_2) + \frac{P(dP)}{K_s} \quad (27)$$

The change of strain energy from e to a is

$$W_a - W_e = \alpha(L_1 + L_2)\int_e^a P(dT) + \frac{1}{K_s}\int_e^a P(dP) \quad (28)$$

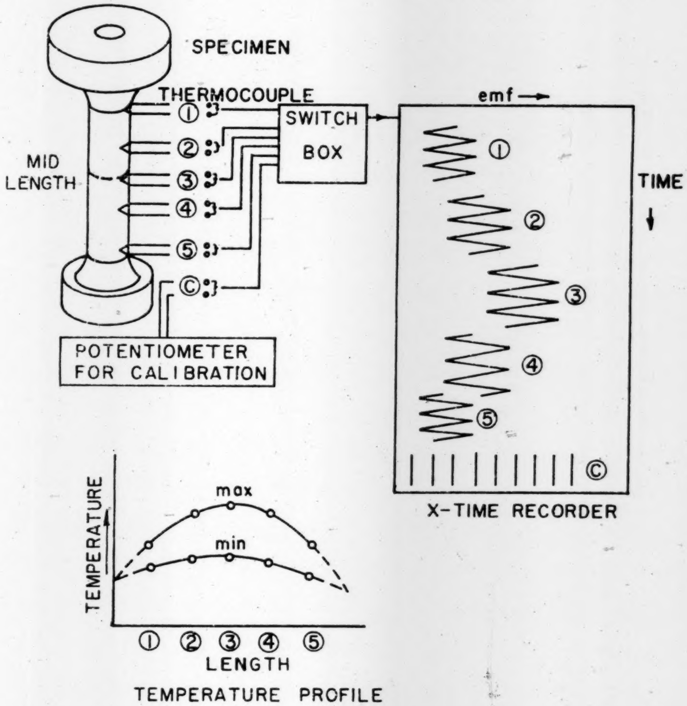

SPECIMEN
THERMOCOUPLE
①
②
③
④
⑤
©
MID LENGTH
SWITCH BOX
POTENTIOMETER FOR CALIBRATION
emf →
TIME
①
②
③
④
⑤
©
X-TIME RECORDER
TEMPERATURE
max
min
① ② ③ ④ ⑤
LENGTH
TEMPERATURE PROFILE

FIG. 5.—Schematic Diagram of Method of Obtaining Temperature Profiles.

The recovered energy on unloading is

$$W_b - W_a = \alpha(L_1 + L_2)\int_a^b P(dT) + \frac{1}{K_s}\int_a^b P(dP) \quad (29)$$

The energy dissipated during the half cycle *e-a-b* is one half of the total plastic strain energy per cycle and is:

$$\frac{\Delta W_p}{2} = \alpha(L_1 + L_2)\left[\int_e^a P(dT) + \int_a^b P(dT)\right] \quad (30)$$

The total plastic strain energy per cycle is therefore the total area contained within the *P-T* loop multiplied by $\alpha(L_1 + L_2)$.

ANALYSIS OF ACTUAL THERMAL FATIGUE TEST

In order to apply the simplified analysis to the actual experiment, one needs to recall the assumptions of that development. It was assumed that there were two distinct lengths having different areas. The plastic strain was assumed to be uniformly distributed in length L_2, and the temperature was independent of length. The material properties were independent of temperature, and the material was elastoplastic.

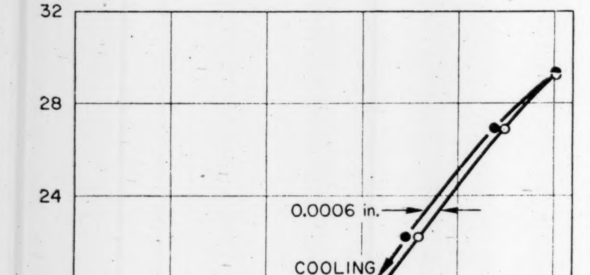


FIG. 6.—Control Thermocouple Output Versus Total Thermal Strain.

General:

A typical thermal fatigue specimen is shown in Fig. 5 with the measured temperature profiles at the maximum and minimum temperatures. The total unrestrained thermal movement δ_T° may be obtained by measuring the displacement of the end plates and is equal to the area between the profiles multiplied by α:

$$\delta_T^\circ = \int_0^L \int_{T_{\mathrm{min}}}^{T_{\mathrm{max}}} \alpha(dT)\, dx \ldots. (31)$$

Wyatt and Jordan (2) have experimentally verified this equation in numerous tests.

For the specimen shown in Fig. 5 the total mechanical strain range obtained from Eq 5 is:

$$-\Delta\delta_m = \int_0^L \int_{T_1}^{T_2} \alpha\, dT\, dx + \frac{\Delta P}{K_s} \ldots (32)$$

The value P/K_s is equal to δ_s and is the deflection of the restraint system. The value of δ_T and δ_s are commonly measured with a dial gage and in this paper are designated by ΔD° and $\Delta D'$ respectively. One is not free to neglect δ_s as being insignificant. The deflection of the restraint constitutes an appreciable unloading effect, thereby making it absolutely necessary that the model contain an element that represents an elastic restraint.

Significance of the Lengths L_1 and L_2.—In the simplified case it was shown that a length greater than gage length contributed to the total thermal strain. This length is $(L_1 + L_2)$. In the actual case it is necessary that an equivalent thermal length be determined. The change of a dial gage reading, ΔD°, during unrestrained thermal expansion and contraction is analogous to the total thermal strain of the model specimen of length $(L_1 + L_2)$ heated uniformly from T_a to T_d,

$$\Delta D^\circ = \alpha(T_d - T_a)(L_1 + L_2) \quad \ldots (33)$$

For the actual case, ΔT_x is not uniform throughout the length and α is usually temperature-dependent. An effective thermal length can be described by the relationship

$$L^\circ = \frac{\Delta D^\circ}{\alpha(\Delta T)_{\max}} \quad \ldots\ldots\ldots (34)$$

and the product of $(\alpha)(L^\circ)$ describes the gradient of thermal strain to temperature change.

Frequently the relationship of thermal strain to the temperature at midlength (where the control thermocouple is located) is nonlinear and is not a single

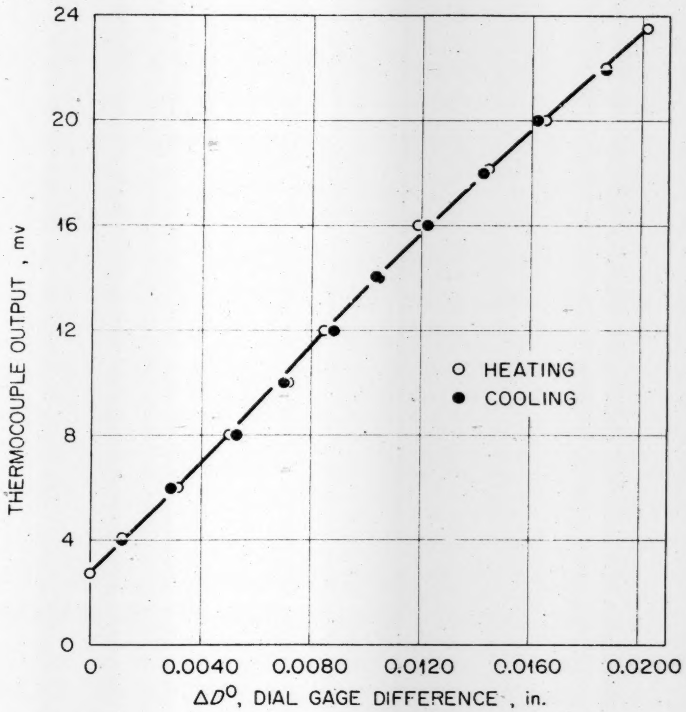


FIG. 7.—Average Thermocouple Output Versus Total Thermal Strain.

valued function. Figure 6 illustrates this condition. The length of the specimen is different for equal values of temperature on heating and cooling. What is needed is a transducer that will give a signal which can be directly related to the amount of thermal strain at any instant during the cycle.

A satisfactory transducer of the instantaneous thermal strain is obtained if several thermocouples are attached to the specimen and located at the centers of equal segments of the specimen length as shown in Fig. 5. The average electromotive force is a convenient sensor of the average temperature of the total length of the specimen. A plot of the average electromotive force versus dial gage difference is shown in Fig. 7. While this relationship of average electromotive force to thermal strain is not always linear, it is a closed loop. From this relationship, the thermal strain component can be determined at any instant during the cycle. The product of $(\alpha)(L^\circ)$ is represented by the tangent to the curve shown in Fig. 7. Moreover, the plot of average electromotive force to thermal strain describes the functional relationship of average temperature (that is, average electromotive force) of the specimen to the thermal strain:

$$(\text{average electromotive force})(\alpha)(L^\circ) = \varphi(\delta_T)$$

The particular value of L° is not as important as the relationship that $(\alpha)(L^\circ)$ represents. If it is desired to evaluate L°, several choices are possible. The thermal coefficient is usually temperature-dependent and $(\Delta T)_{\max}$ can be taken from the control thermocouple or the average electromotive force of the specimen. As a general rule, α should be evaluated at the mean temperature and $(\Delta T)_{\max}$ should be determined from the extremes of the average electromotive force cycle.

The value of L° is always greater than the length of the straight section and is nearly equal to the distance between the massive ends of the specimen. It is absolutely necessary that the model specimen be composed of two different segments because in a real specimen (1) the fillets are larger in area and constitute about one third of the total length; (2) the fillets cycle in temperature and contribute to the total thermal strain; and (3) the fillets sustain little, if any, plastic strain.

From a load-temperature loop of the actual specimen the total plastic strain can be determined. The internal width of the P-T loop at zero load is $(T_b - T_e)$. The length $(L_1 + L_2)$ is simply L° as described above. The length L_2 of the model is the length sustaining a uniformly distributed plastic strain. In the actual case the unit plastic strain is not uniformly distributed; however, the total plastic strain is given by

$$-\Delta\delta_p = \alpha(T_b - T_e)L^\circ \ldots\ldots (35)$$

If the length sustaining plastic strain is not known, a secondary strain measuring system must be employed to determine the average unit plastic strain value of the center 1 in. of the specimen length. An effective gage length L' defines the relationship of the total plastic strain of Eq 35 to the average $\Delta\epsilon_p$ value determined by the secondary measuring system:

$$L' = \frac{\Delta\delta_p}{\Delta\epsilon_{p\ \text{avg}}} \ldots\ldots\ldots\ldots (36)$$

The value of L' represents L_2 of the simplified case and is defined as the ratio of the total plastic strain of the specimen to the average unit plastic strain of a short central length of the specimen. The temperature is almost uniform in this length, and a post-test examination of the specimen will indicate if the plastic strains were nearly uniform.

To obtain the average $\Delta\epsilon_p$ of a central length a sensitive strain measuring system, preferably an optical one, should be used to measure the total geometric displacement, δ_c, of markers placed at the ends of the length. During the temperature cycle the displacement δ_c is the thermal strain plus the mechanican strain:

$$\delta_c = \delta_T + \delta_m \qquad (37)$$

Because the gage length is unity,

$$\Delta\delta_c = \alpha\Delta T + \frac{\Delta\sigma}{E} + \Delta\epsilon_{p\ \text{avg}} \qquad (38)$$

If two points of the P-T cycle are conveniently chosen at the zero load condition on heating and cooling, the elastic component of Eq 38 is zero, and

$$\Delta\epsilon_{p\ \text{avg}} = -\alpha(T_b - T_e) + \Delta\delta_c \Big|_b^e \qquad (39)$$

The effective gage length L' for the test is:

$$L' = \frac{\alpha(T_b - T_e)L^\circ}{\alpha(T_b - T_e) - \Delta\delta_c \Big|_b^e} \qquad (40)$$

That L' and L° are not equal should be obvious. The length L' is not a constant for all tests. It is dependent on the maximum temperature, the temperature difference, the material, and the specimen geometry. To determine L' for one test and apply it to all test conditions will produce an erroneous slope to the log $\Delta\epsilon_p$ versus log N_f curve.

A very simple method for approximating L' can be employed without special strain measuring devices. On specimens sustaining large values of cyclic plastic strain the machined surface is disturbed by the deformation. The length of this deformed region can be easily measured and is approximately equal to L'.

The value of L' decreases with increasing values of $\Delta\epsilon_p$ and maximum temperature. Demers (3) has shown the value of L' to be stable during the test period and to increase slightly for increasing wall thickness. For the wall thickness investigated (0.020 to 0.060 in.) only a slight change of the temperature profile was noted. For the conventional specimen design the effect of using values of both L' and L° in error in the order of 0.25 in. will produce an error of about 25 per cent in the computed plastic strain value.

The tubular specimen design is sometimes susceptible to geometric instabilities that make it unsuitable as a standard, or reference, test specimen. Tubular specimen data are conservative, however, and this is not without merit. However, the influence of the specimen geometry must be considered if thermal fatigue data are to be appreciated.

Method I, Equation 10.—The plastic strain range by Method I is:

$$\Delta\epsilon_p = -\alpha(\Delta T)_0 \frac{L^\circ}{L'} \qquad (41)$$

where $(\Delta T)_0$ is the width of the load-temperature loop at zero load. It is not necessary to state the value of α explicitly. Equations 41 and 34 can be combined to read

$$\Delta\epsilon_p = -\frac{(\Delta T)_0}{(\Delta T)_{\max}} \left(\frac{\Delta D^\circ}{L'}\right) \qquad (42)$$

Two of the objections to the thermal fatigue test are that the modulus of elasticity, the thermal coefficient, and the strength of the material vary with temperature and that the thermal coefficient varies with stress. Thus, say the critics, a large uncertainty is included in any calculated value of $\Delta\epsilon_p$. To determine $\Delta\epsilon_p$ by Eq 42, the values of E, α, A_1, A_2, L°, K_s, and the calibration factor of the load cell are not required. It is trivial to show that α is stress-dependent for the plastic strain is calculated by measuring the width of the *P-T* loop at the two zero stress conditions.

An analogy of Eq 42 can be described in three steps: (1) the length of an unloaded bar is measured; (2) the bar is heated (or cooled) in a restrained condition and sustains plastic flow; and (3) the bar is unloaded and the length remeasured. Equation 42 provides a means for calculating the amount of plastic flow that results from restrained thermal loading, without regard to the variation of properties during that period. If only one thermocouple is used as a sensor of thermal strain the electromotive force versus thermal strain relationship must be a closed loop on heating and cooling. The temperatures of zero load on heating and cooling are near the mean temperature of the test; therefore if an optical strain gage and α are used to determine L', α at the mean temperature is the better choice. Thus, the assumption of α being independent of stress and temperature is seen to be reasonable.

Method II, Equation 13.—Equation 13 can be rewritten to allow calculation of $\Delta\epsilon_p$ by

$$-\Delta\epsilon_p = \alpha(\Delta T)_{\max}\frac{L^\circ}{L'} + \frac{\Delta\sigma_1}{E}\frac{L_1}{L'} + \frac{\Delta\sigma_2}{E} - \frac{\Delta P}{K_s L'} \quad (43)$$

The necessity of evaluating ΔP, E, L_1, A_1, and K_s add additional uncertainties that are not included in Method I. The value L_1 can be approximated by $(L^\circ - L')$. The omission of L°/L' will produce an error in the slope and location of the log $\Delta\epsilon_p$ — log N_f curve. Because many materials relax at elevated temperature, the relaxed or final values of stress and load at the maximum and minimum temperatures must be used. The approximation of Eq 43 by

$$-\Delta\epsilon_p = \alpha(\Delta T) - \frac{\Delta\sigma}{E} \quad (44(a))$$

in many cases is unnecessarily conservative (that is, gives too low a value of $\Delta\epsilon_p$). Equation 44(*a*) has been commonly used to determine the plastic strain range of the thermal fatigue test (6). A comparison of Eqs 44(*a*) and 43 should illuminate why the results can contain gross errors. Another variation of Eq 44(*a*) found implied in the literature (7) is

$$-\Delta\epsilon_p = \alpha(\Delta T) - \frac{\Delta D'}{L^\circ} \quad (44(b))$$

Considerable error can also accompany its use.

Method III, Equation 18.—Equation 18 can be applied to the actual case by

$$-\Delta\epsilon_p = \frac{P_f - P_c}{L'}\left(\frac{L^\circ - L'}{A_1E_1} + \frac{L'}{A_2E_2} + \frac{1}{K_s}\right) \quad (45)$$

The evaluation of an effective area A_1 is inconvenient, and a constant temperature line must be selected that intersects the linear portion of the heating and cooling curves. Equation 45 will be applicable only to tests where the plastic strain values are small. To neglect the deflection of the restraint and reduce Eq 45 to

$$-\Delta\epsilon_p = \frac{\Delta\sigma_2}{E}\Bigg]_c^f \quad (46)$$

is unjustified. A calculation of $\Delta\epsilon_p$ by Eq 46 will generally lead to errors of 100 per cent or greater. Two points on an isothermal line of the *P-T* loop do not have equal strain values in the stress-strain graph, as indicated in Fig. 3. Equation 46 was proposed as a method for calculating the plastic strain range (8). Early results obtained from its use were so questionable that further use was not recommended. The idea underlying its proposal is valid and the method derived from the model (Eq 45) is quite useful and gives more consistent results than Eq 43.

Method IV, Autographic Actual P-T Loop:

The actual load-temperature hysteresis loop is best obtained by connecting in parallel the outputs of five thermocouples as shown in Fig. 5 and plotting the average of the five thermocouple electromotive forces versus load. Most *X-Y* recorders will accept d-c millivolt inputs without additional instrumentation. The load cell output, suitably amplified, is used to drive the *Y*-axis; the average thermocouple output supplies the *X*-axis signal. Thus an autographic *P-T* cycle is readily obtained. Any one of the methods already described can be used with this loop to determine the plastic strain range, the stress range, the mechanical strain range, and the plastic strain energy.

TABLE 1.—THERMAL FATIGUE DATA FOR HASTELLOY N.
Specimen H-36, Cycle No. 8

Temperature limits at control thermocouple, 120 to 1310 F
Cycle period, 150 sec

$$\left.\begin{aligned}\alpha &= [0.00362\ T + 5.46]\ 10^{-6}\text{ per deg F}\\ E &= [0.012\ T + 27.5]\ 10^{6}\text{ psi}\end{aligned}\right\}\ 70\text{ F} < T < 1600\text{ F}$$

$K_s = 1.11 \times 10^6$ lb per in.
$A_2 = 0.0755$ sq in.
$A_1 \approx 2.5\ A_2$ (approximate)
$L' = 1.25$ in. (length of deformed surface on specimen)
$\Delta D^\circ = 0.0202$ in.
$\Delta D' = 0.0078$ in.

From average electromotive force *P-T* loop (see Fig. 8):
$T_{\min} = 273$ F
$T_{\max} = 1183$ F
$T_e - T_b = 81.2$ F
$dP/dT = 10.04$ lb per deg F (measured)
$P_a - P_d = 8650$ lb
$P_f - P_e = 810$ lb
$L^\circ = 2.75$ in.

Calculated values of plastic strain range, $\Delta\epsilon_p$, μin. per in.:

	Derived from Model			Reported in Literature		
	Method I, Eq 42	Method II, Eq 43	Method III, Eq 45	Method A, Eq 44(*a*)	Method B, Eq 44(*b*)	Method C, Eq 46
$\Delta\epsilon_p$	1440	720	1370	3480	5670	565

Slope of (average) *P-T* loop at zero load = 10.05 lb per deg F; calculated from Eq 22.

Equation 5 is a simple yet powerful tool for analyzing the thermal fatigue test. The geometric strain, δ_s, is the deflection of the end plates and is a linear function of load. The elastic component of the mechanical strain is also a linear function of load. The thermal strain, δ_T, is the total thermal strain of the specimen. The average of the electromotive forces of several thermocouples equally spaced along the length is the sum of the elastic and plastic strains in the specimen. Therefore if δ_T can be plotted versus load, elastic loading and unloading will produce a linear, closed loop.

Experimental Results:

The purpose of this paper is not to publish experimental results. A few examples of experiment, however, may demonstrate the validity of the model.

The first example is that of a conventional test on a specimen of Hastelloy N, a nickel-base alloy. The data for cycle number 8 is shown in Table 1. A graph of the load versus average temperature for cycle number 8 is shown in Fig. 8. The graph of the average electromotive force versus thermal strain for the unrestrained condition is shown in Fig. 7. Methods I and III based on the model give results which show good agreement. The approximations of the values A_1 and L_1 have a much greater effect on Method II than on Method III and seriously limit Method II's usefulness. Methods that have been reported

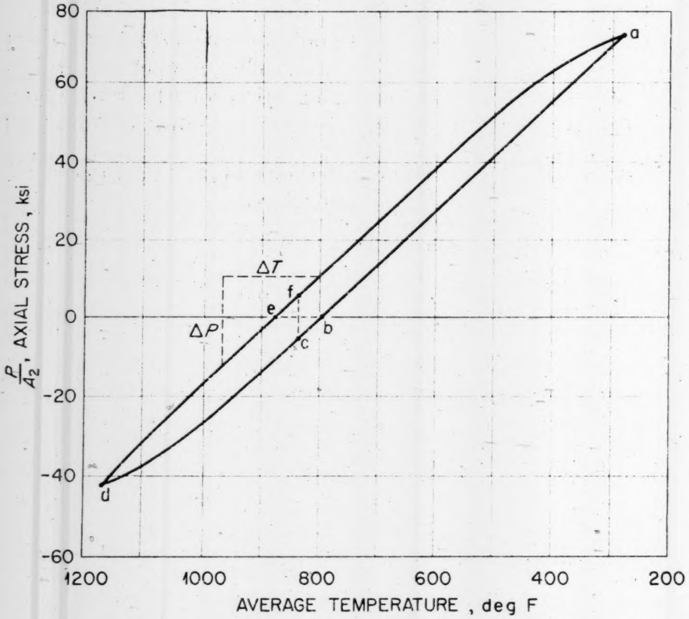


FIG. 8.—Stress-Temperature Loop, Specimen H-36, Cycle No. 8.

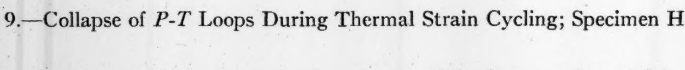


FIG. 9.—Collapse of P-T Loops During Thermal Strain Cycling; Specimen H-36.

earlier in the literature show widely divergent results. The slope at zero load of the *P-T* loop shows excellent agreement with that predicted by Eq 22. The plastic strain energy of this loop shows good agreement with that calculated from the stress range, plastic strain range, and strain hardening exponent. The collapse of the *P-T* loop is shown in Fig. 9 and the decrease of the plastic strain range is shown in Fig. 10. Because the plastic strain is not constant, Eq 47 was used to determine the average value:

$$\Delta\epsilon_{p\ \mathrm{avg}} = \frac{\Delta\epsilon_{p1}}{(N_f)^B(1 - B)} \quad \ldots\ldots (47)$$

where $\Delta\epsilon_{p1}$ is the value of the plastic strain range on the first cycle, B is the logarithmic slope of $\Delta\epsilon_p$ to N, and N_f is the number of cycles to failure. In the example cited $\Delta\epsilon_{p\ \mathrm{avg}}$ is 116 microunits. By selecting the most sensitive millivolt scale for the temperature axis on the *X-Y* recorder and determining an enlarged *P-T* loop at zero load it was possible to determine plastic strain range values in the order of 70 microunits.

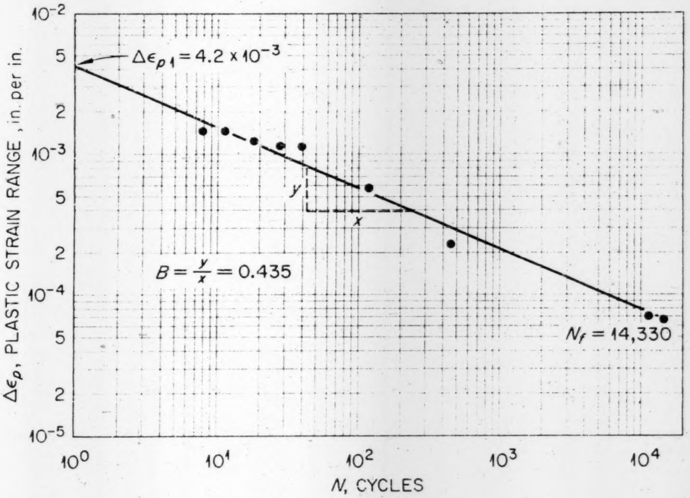


FIG. 10.—Decrease of Plastic-Strain Range During Thermal-Strain Cycling; Specimen H-36.

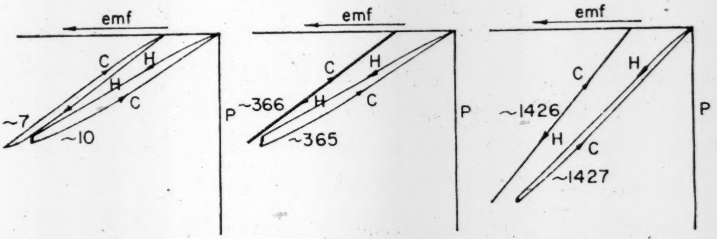


FIG. 11.—Difference Between Average Temperature (*Left*) and Control Temperature (*Right*) *P-T* Loops; Specimen H-38.

When the plastic strain range values are very small it is necessary to use the average temperature *P-T* loop. The difference between the average temperature and control temperature *P-T* loop for another specimen is indicated in Fig. 11. In this very small plastic strain region the average plastic strains determined from the control *P-T* loops were greater than those determined from the average temperature *P-T* loops by factors of from 3 to 7.

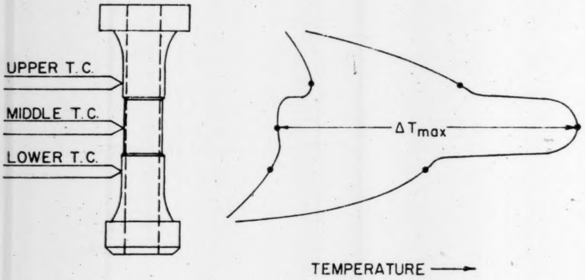


FIG. 12.—Maximum and Minimum Temperature Profiles; Duplex Specimen, Resistance Heating Rapid Cycling.

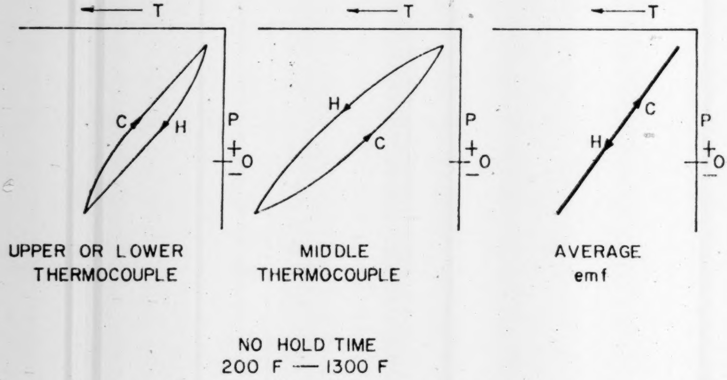


FIG. 13.—*P-T* Loops of Duplex Specimen, Rapid Cycling.

A second example is that of a duplex specimen of the same material under similar test conditions. This type of specimen design, shown in Fig. 12, was selected because it more nearly conforms to the geometry of the model, and more severely exaggerates the lack of uniform temperature distribution. Initially the specimen was rapidly heated and cooled, the frequency being about 1 cpm. Because of its large thermal mass and low resistance, the large end section thermally lags the temperature cycle of the central gage portion. A plot of the temperature profile is shown in Fig. 12. The *P-T* loops from the upper, central, and average of all of the thermocouple electromotive forces versus load are shown in Fig. 13. Note the inverted loop (*CCW*) of the central section *P-T* loop. The upper section and central section *P-T* loops are both open loops and deceptively indicate that a plastic strain was sustained on each cycle. After 20,000 cycles of constrained thermal cycling no disturbance was apparent on the specimen surface and externally the specimen indicated that little, if any, plastic strain resulted from the thermal strain cycling. The average electromotive force *P-T* loop also gave this indication in that the loop was a closed straight line as shown in Fig. 13.

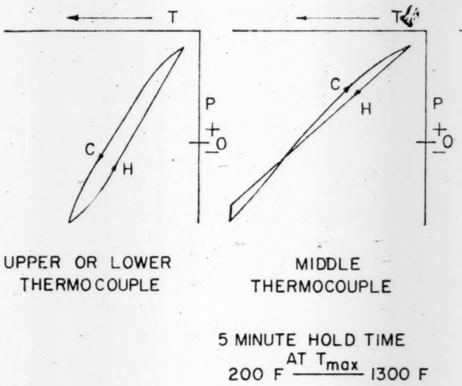

T
P
+
0
−
C
H
UPPER OR LOWER THERMOCOUPLE
MIDDLE THERMOCOUPLE
AVERAGE emf
5 MINUTE HOLD TIME
200 F AT Tmax 1300 F

FIG. 14.—*P-T* Loops of Duplex Specimen, Hold Time at Maximum Temperature.

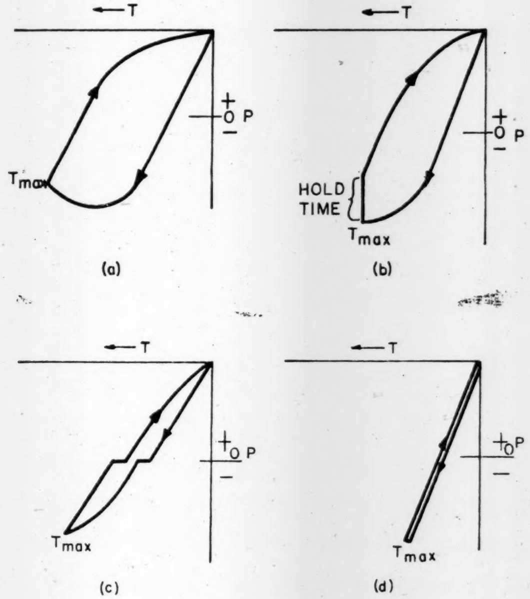
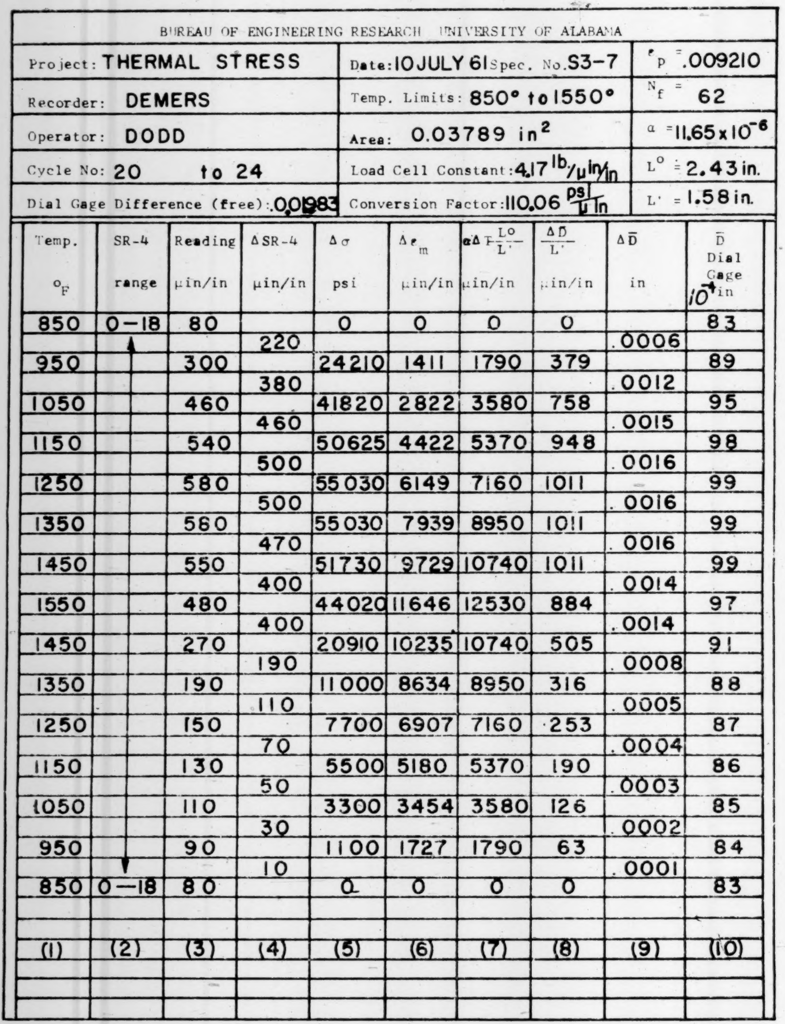

T
+
0 P
−
Tmax
HOLD TIME
(a)
(b)
(c)
(d)

FIG. 15. Typical *P-T* Loops.

The test conditions were then changed to include a 5-min hold time at 1300 F. The control system operates to maintain a constant temperature at the midlength of the specimen. This, of course, does not imply a constant total thermal strain during the hold time. Several minutes are required for the temperature profile to reach equilibrium. Under these conditions the central section *P-T* loop crossed over, the upper section *P-T* loop indicates little plastic strain, but the average electromotive force *P-T* loop indicates plastic strain in the order of 1 per cent per cycle as shown in Fig. 14. During cycling the specimen surface showed severe deformation bands, the diameter increased slightly and after 425 cycles the specimen failed.



BUREAU OF ENGINEERING RESEARCH UNIVERSITY OF ALABAMA

Project: THERMAL STRESS
Date: 10 JULY 61 Spec. No. S3-7
ϵ_p = .009210
Recorder: DEMERS
Temp. Limits: 850° to 1550°
N_f = 62
Operator: DODD
Area: 0.03789 in^2
α = 11.65 x 10^{-6}
Cycle No: 20 to 24
Load Cell Constant: 4.17 lb/μin/in
L^o = 2.43 in.
Dial Gage Difference (free): .00983
Conversion Factor: 110.06 psi/μin
L' = 1.58 in.

Temp. °F	SR-4 range	Reading μin/in	ΔSR-4 μin/in	$\Delta\sigma$ psi	$\Delta\epsilon_m$ μin/in	$\alpha\Delta T\frac{L^o}{L'}$ μin/in	$\frac{\Delta\bar{D}}{L'}$ μin/in	$\Delta\bar{D}$ in	$\bar{D}$ Dial Gage 10^{-4} in
850	0–18	80		0	0	0	0		83
			220					.0006	
950		300		24210	1411	1790	379		89
			380					.0012	
1050		460		41820	2822	3580	758		95
			460					.0015	
1150		540		50625	4422	5370	948		98
			500					.0016	
1250		580		55030	6149	7160	1011		99
			500					.0016	
1350		560		55030	7939	8950	1011		99
			470					.0016	
1450		550		51730	9729	10740	1011		99
			400					.0014	
1550		480		44020	11646	12530	884		97
			400					.0014	
1450		270		20910	10235	10740	505		91
			190					.0008	
1350		190		11000	8634	8950	316		88
			110					.0005	
1250		150		7700	6907	7160	253		87
			70					.0004	
1150		130		5500	5180	5370	190		86
			50					.0003	
1050		110		3300	3454	3580	126		85
			30					.0002	
950		90		1100	1727	1790	63		84
			10					.0001	
850	0–18	80		0	0	0	0		83
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)

FIG. 16.—Data Sheet for Thermal Fatigue Stress-Strain Diagram.

The inference to be drawn from this discussion is that the plot of the average electromotive force versus load will be an open loop only if the thermal cycling produces plastic strain in the specimen. Too, the control thermocouple *P-T* loop can be grossly misleading. Most of the published thermal fatigue results have been based on temperature information obtained from the control thermocouple. While the average electromotive force *P-T* loop method does not cure all of the maladies of this test, it is definitely superior to the methods commonly used in the past.

The average electromotive force P-T loop method determines the total plastic strain $\Delta\delta_p$, and only if the length of the specimen sustaining plastic flow is constant, that is, L', will the unit plastic strain be a linear function of the internal width of the loop. The two most common causes of variable L' during testing are gross changes in the specimen geometry (bulging) and severe strain hardening. Both of these phenomena are readily detected. In the test just described the material sustained a 120,000 psi stress change and a 1100 F temperature change, yet for the no hold-time condition a closed linear P-T loop (average electromotive force) was obtained. This fact would seem to suggest that temperature or stress-dependence, or both, of α and E are not significant impediments to the analysis of the thermal fatigue test and do not introduce any appreciable nonlinearities to the elastic behavior. Experiment does not substantiate the critics' objection that the temperature and stress-dependence of material properties introduce gross uncertainties to this type of test.

The multiple thermocouple load-temperature loop is strongly recommended. Much information can be gleaned from these diagrams and one or more of the equations developed can be used to obtain the test parameters. The P-T loop shown in Fig. 15(*a*) indicates the decrease of elastic strength near the maximum temperature. The relaxation of elastic stress that occurs during the hold time at the maximum temperature is shown in Fig. 15(*b*). Slack in the clamping mechanism is apparent in the loop of Fig. 15(*c*) and elastic cycling is shown in Fig. 15(*d*). Strain hardening and strain softening can be easily detected, and the area of the P-T cycle is proportional to the plastic strain energy dissipated per cycle. The plastic strain energy, the plastic strain range, the mechanical strain range, and the stress range can all be determined from the P-T loop.

The Stress-Strain Diagram:

A stress-strain hysteresis loop can be plotted if digital data are obtained from the load cell, average temperature of the specimen, and a dial gage that measures the displacement of the restraint. A typical data sheet is shown in Fig. 16. The temperature cycle is divided into convenient increments, and at these temperatures, readings are obtained from the load cell indicator and dial gage. The stress change over the increment is:

$$\Delta\sigma_2 = \frac{(\Delta SR\text{-}4)(CF)}{A_2} \quad \ldots\ldots\ldots (48)$$

The change in load cell output is ΔSR-4, (CF) is the calibration constant of the load cell in pounds per microunit, and A_2 is the minimum cross-sectional area of the gage portion of the specimen.

Over the same interval of temperature the unit mechanical strain change is calculated from the temperature increment ΔT and the increment of dial gage change, $\Delta D'$.

$$-\Delta\epsilon_m = \alpha(\Delta T)\frac{L^\circ}{L'} - \frac{\Delta D'}{L'} \quad \ldots\ldots (49)$$

In this example the elastic strain in the fillets was neglected and ΔT at midlength was used rather than ΔT from the average electromotive force sensor. The data of columns 1, 2, 3, and 10 are used to calculate the stress and strain values of columns 5 and 6 of Fig. 16. If one assumes an arbitrary zero of the coordinate system to be at the minimum temperature, all of the loop will appear in the third quadrant. The data of Fig. 16

were used to plot the hysteresis loop of Fig. 17. The slope of the σ-ϵ diagram at zero load is about 20×10^6 psi which shows rather good agreement with the modulus of elasticity value at 1200 F.

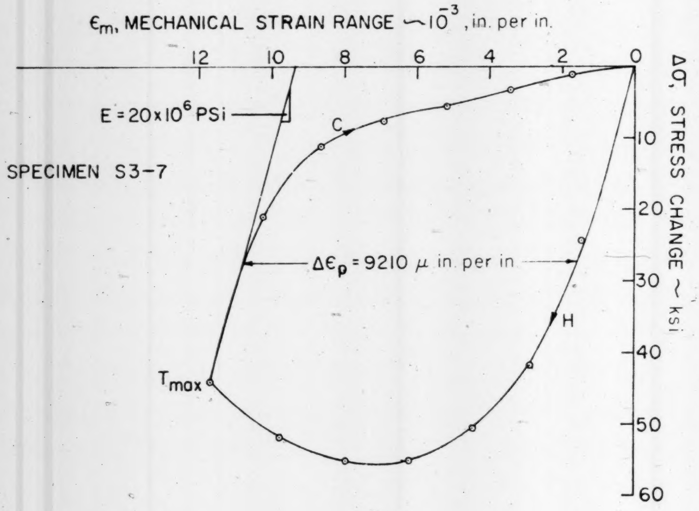


FIG. 17.—Thermal Fatigue Stress-Strain Diagram.

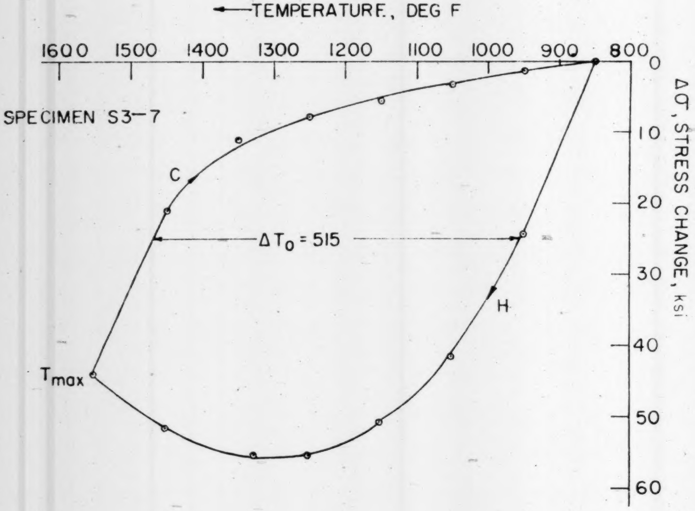


FIG. 18.—Thermal Fatigue Stress-Temperature Loop.

A stress-temperature loop constructed from the data is shown in Fig. 18, and a 515 F maximum width is observed. The $\Delta\epsilon_p$ values evaluated from several methods are:

σ-ϵ loop	0.009210	Average 0.008824
Method I	0.008620	
Method II	0.008594	
Equation 44(*a*)	0.005410	

Method III is applicable only in tests of materials having large values of elastic strength. It is not used for this example because of the lack of sufficient elasticity. The autographic *P-T* loop method is the analog equivalent of Method I. The results obtained from Eq 44(*a*) are included to illustrate the disparity of values. For small values of $\Delta\epsilon_p$, Eq 44(*a*) yields much smaller values than Methods I or II. There have been occasions reported where Eq 44(*a*) was used and negative $\Delta\epsilon_p$ values were obtained (4). This is easily explained. The value $\Delta\epsilon_p$ is equal to $\alpha(\Delta T)(F) - (\Delta\sigma_2)/E$. The constraint factor F is greater than 1 and for the example cited is 1.43. For a material with sizable elastic strength and a test producing little or no $\Delta\epsilon_p$, it is obvious that $(\Delta\sigma_2)/E$ could be larger than $\alpha(\Delta T)$ if F is assumed to be unity rather than its actual value.

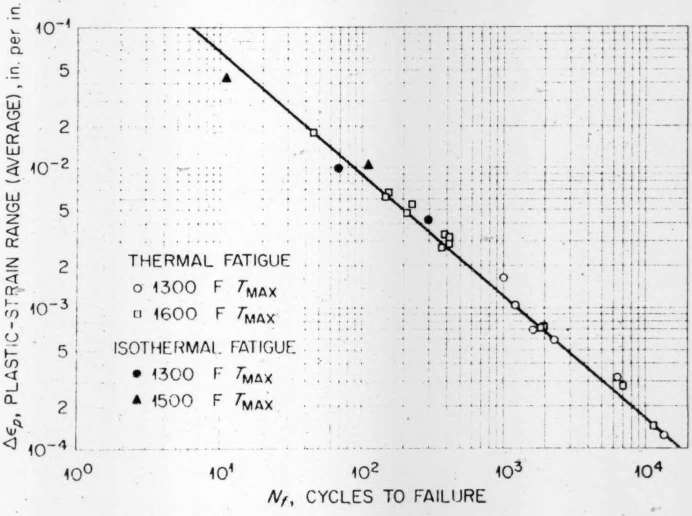


FIG. 19.—Plastic-Strain Range Versus Cycles to Failure, Hastelloy N.

An autographic stress-strain loop can be obtained if the difference of the output of a displacement transducer (in lieu of a dial gage) and the output of an average temperature sensor are used to drive the strain axis. During free cycling the outputs of the temperature and dial gage transducers are made to be equal and opposite. In essence, the mechanical strain δ_m of Eq 5 is being plotted versus load.

Finally the results of several thermal fatigue tests of Hastelloy N are shown in Fig. 19. These specimens evidenced only slight changes in geometry (bulging, and so on) during the test. Isothermal strain cycling results (1300 F and 1500 F) are also included in the figure and show excellent agreement to the thermal strain cycling data (1300 F and 1600 F $T_{\max}$). A few test data are also included in the thermal fatigue results that were obtained from duplex type specimens. Significant changes of specimen geometry are not discernible in the results. If the calculation of $\Delta\epsilon_p$ for the thermal fatigue experiment is based on methods published in the literature large scatter of the data is obtained and the thermal fatigue results do not agree with the isothermal. Perhaps other disagreement of isothermal and thermal fatigue data observed and reported is only apparent because of the deficiencies of the earlier methods of analysis.

SUMMARY

The purpose of this paper has been to describe a thermal fatigue model that would adequately fit the conventional thermal fatigue test; to analyze the model; and to apply the analysis to the actual test. Several examples of actual tests were described in which the methods derived were used to obtain test data. The results of the several methods were compared and these comparisons lend confidence to the adequacy of the model. It has been demonstrated how the average electromotive force of multiple thermocouples will indicate the total thermal strain of the specimen length at any instant of time during the cycle. The load-temperature loop obtained from a plot of the average electromotive force versus load is probably the most significant record of the test and can be used to obtain the total plastic strain, the total mechanical strain, the stress range, and the total plastic strain energy of the cycle. Several of the common $\Delta\epsilon_p$ equations found in the literature were compared to the equations derived from the model. These comparisons illuminate why the deficiencies of the earlier methods exist and show why the data from some of these earlier tests do not agree with service life and why in certain cases such large differences between isothermal and thermal fatigue data were obtained.

It is concluded that the thermal fatigue test can be conducted and the mechanical property parameters of the test can be determined with a reasonable degree of accuracy and confidence. The data of the thermal fatigue test are probably more indicative of the actual resistance of the material to restrained thermal cycling than isothermal strain cycling data.

Acknowledgments:

The analysis presented in this report was, in part, performed at the University of Alabama for a research project sponsored by the Metals and Ceramics Division of the Oak Ridge National Laboratory.[4] Appreciation is expressed for this support and to the members of the Division who have patiently assisted and guided this work.

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[4] Operated by the Union Carbide Corp. for the U. S. Atomic Energy Commission.

AN APPARATUS FOR THE DIRECT DETERMINATION OF DUCTILE-BRITTLE TRANSITION TEMPERATURES ON WIRE OR STRIP SPECIMENS*

By D. L. Wood[1] and J. H. Westbrook[1]

Synopsis

The measurement of ductile-brittle transition temperatures normally involves destructive isothermal testing of a large number of specimens. A device has been built which enables the determination to be made with a single test on one specimen. A wire or strip specimen is continuously bent around a rotating drum while the oil bath in which the equipment is immersed is lowered in temperature. The transition temperature is defined as that temperature at which the specimen will no longer tolerate bending to a strain determined by the diameter of the drum and the thickness or diameter of the specimen. The test has been applied to a study of the effects of such metallurgical variables as strain rate, grain size, and composition in a variety of materials.

Many materials experience an abrupt change from ductile to brittle failure when the conditions under which they are stressed are varied. Such a transition may occur with a change in temperature strain rate or state of stress and has been found in a wide variety of materials: body-centered cubic and hexagonal close-packed metals, covalent metalloids such as silicon and germanium, intermetallic compounds, and organic polymers. (Face-centered cubic metals are a notable exception.) Above the transition, yielding occurs and some appreciable plastic deformation precedes a shear-type rupture. Below the transition, little or no deformation occurs prior to fracture, which may proceed by cleavage or intergranular rupture. Such a drastic change in mechanical behavior has obvious practical consequences and is also of fundamental interest in studies of flow and fracture.

For purposes of prooftesting a part or material that is to be subjected to known service conditions, it may sometimes be necessary to examine the response of some ductility parameter to one or more particular variables over a considerable range of the variable. On the other hand, in many practical cases and in most fundamental studies only the critical value of the test variable at which the ductile-brittle transition occurs is sought. The substance of the research in such cases will lie in assessing the dependence of this transition parameter upon various material characteristics. Furthermore, the established equivalence (in this sense) of the three variables—temperature, strain rate, and stress state—permits the experimenter to choose the most convenient variable to work with in characterizing the ductile-brittle transition. Such considerations have led to the frequent use of a "transition temperature" as a parameter in studies of fracture.

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* Presented at the Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] General Electric Research Laboratory, Schenectady, N. Y.

Typical curves illustrative of a ductile-brittle transition are shown in Fig. 1. Curve *A* represents ideal behavior while curve *B* is more nearly representative of a real material. The ordinate axis *P* represents the ductility parameter and the abscissa *Q*, the test variable. Even when the type of test is specified there may be a further choice in *P*, the ductility parameter—for example, reduction of area, fracture appearance, energy absorption, fracture strength, or strain. The types of test used to determine such curves include tension tests, torsion tests, and bend tests; strain rates may vary from very slow as in conventional tension machines, to medium in standard impact types, to relatively high in pneumatic or ballistic impact devices. A familiar specific example of a test that can yield data of the type shown in Fig. 1 would be the Charpy impact test where energy absorption is measured as a function of temperature. When the temperature is reduced to some low temperature where the transition to brittle failure occurs, a sharp reduction in energy absorption is found since much less energy is required for brittle fracture than for ductile fracture. An analogous change would have been observed if the strain rate had been raised sufficiently or the stress state made more highly triaxial.

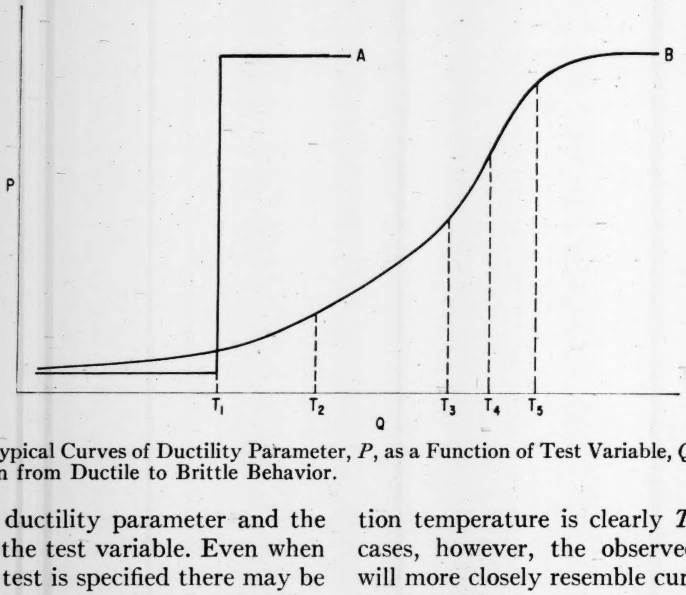

FIG. 1.—Typical Curves of Ductility Parameter, *P*, as a Function of Test Variable, *Q*, Illustrating the Transition from Ductile to Brittle Behavior.

Frequently the behavior of real materials approximates closely the schematic curve, *A*, in Fig. 1, for which the transition temperature is clearly T_1. In some cases, however, the observed behavior will more closely resemble curve *B*. Here the transition temperature may be: T_2, the temperature at which the ductility parameter has attained some arbitrary low value; T_3, the temperature where the parameter is halfway between the upper and lower levels; T_4, the temperature of the inflection point on curve *B*; or T_5, the temperature where the ductility parameter is detectably below its upper level. Notwithstanding a certain degree of arbitrariness in the definition of a transition temperature, little practical difficulty is encountered in its use since for a given type of material the shape of the curve is usually not altered much by changes in the material variables.

All of the usual methods of determination of transition temperatures involve rather time-consuming and tedious testing of large numbers of specimens. They are wasteful of material and, more importantly, variation in material from specimen to specimen necessarily contributes to the scatter and thus limits the utility of such tests in studies of the effects of metallurgical variables on the transition temperature. A test method which would measure a transition temperature directly with one test on a single specimen would have numerous advantages. Consumption of test material would be greatly reduced, increased efficiency would give feasibility to studies of broader scope, and the effects of material variables could be more precisely documented. An apparatus capable of such measurements has been built, and its usefulness demonstrated by documenting the effects of some metallurgical variables on the transition temperature of two materials.

EXPERIMENTAL

Materials:

Tungsten was chosen as a test material primarily because it represents a class of economically important materials in which the measurement and control of ductility is of utmost importance and one for which the ductile-brittle transition is particularly marked. The material used was GE No. 218 lamp filament stock of 0.040-in. diameter. Intermetallic compounds were of interest in that they constitute a material class in which brittle behavior persists to considerably higher temperatures than those normally observed for metals and alloys and thus provide a challenging group of materials for studies of transition temperature. AgMg is a prototype of the broad class of highly ordered intermetallic compounds. Its moderate melting point, broad homogeneity range, and simple crystal structure have led to its selection as the test material for several recent studies. Extruded AgMg wires of 0.040-in. diameter were available from prior studies by the authors of the effects of metallurgical variables on the mechanical behavior of this compound (**1**,**2**).[2]

Apparatus:

The apparatus, illustrated schematically in Figs. 2 and 3, is designed to bend a wire or strip specimen at a constant rate around a drum of uniform diameter while the temperature of the specimen is being decreased. The "transition temperature" is defined as that temperature at which the specimen will no longer tolerate bending at a given rate to the fixed strain determined by the diameter of the drum and the diameter or thickness of the specimen. This transition temperature is defined in the same way as T_2 of Fig. 1.

The specimen is fastened both to the drum and to the sliding guide by a specimen grip. For wire specimens a grooved split plate type grip was used; a simple grip modification would be necessary for strip specimens. Particular care was taken to fair the groove of the drum grip gradually to the drum radius so that premature failure did not occur at the clamping point on the drum. Specimen lengths of from 2½ to 9 in. can be accommodated. Spring loading of the sliding guide as well as an upper guide roll near the drum are provided to keep the test specimen straight.

Variation in relative strain rate in eleven steps from 1 to 180 is accomplished by various combinations of motor speed and drive gears. At the slowest rate the specimen is bent around the drum at 1 linear inch per 18 min; at the fastest rate 1 linear inch of specimen is bent every 6 sec. Further variation could be accomplished either by changing the diameter of the drum (2-in. diameter in the present case) or the thickness of the specimen (0.040-in. diameter in the present case) if these parameters were changed proportionately so as to maintain a constant strain.

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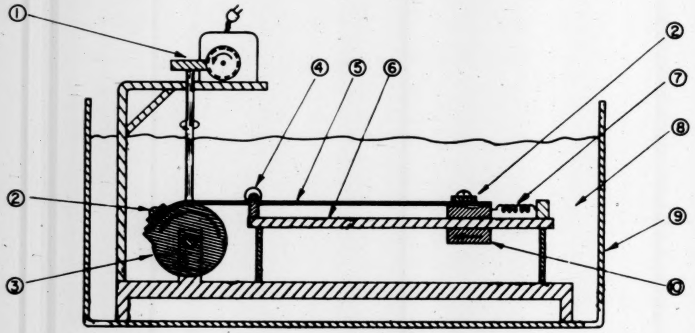
[2] The boldface numbers in parentheses refer to the list of references appended to this paper.



1, power assembly
2, specimen grip
3, rotating drum
4, guide roll
5, specimen
6, slide rod
7, spring load
8, oil bath
9, tank
10, sliding guide

FIG. 2.—Schematic Diagram of Bend Tester.

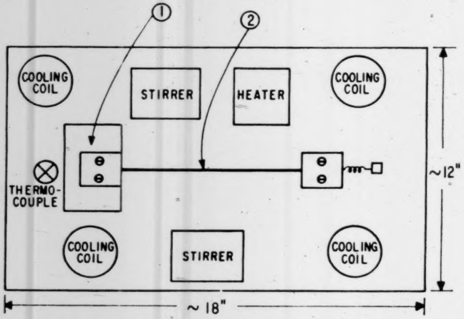


FIG. 3.—Disposition of Heater, Cooling Coils, Stirrers, and Thermocouple in Oil Bath: (*1*) Rotating Drum; (*2*) Specimen.

Control of temperature is obtained by immersing the mechanical apparatus in a silicone oil bath. The bath is heated initially to a temperature sufficient to permit bending of the specimen without fracture. After this condition has been established, the drum is set in continuous rotation and cooling rates faster than normal are obtained by forced water cooling through a number of copper coils immersed in the bath. As the bath and specimen cool, bending is continued until fracture occurs; the temperature of the bath at the time of fracture is measured with a thermocouple and recorded. Some examples of cooling rates available in the present apparatus are shown in Table I.

Figure 4 shows the temperature-time recorder curves for an AgMg compound, with the temperature at which fracture occurred marked with an encircled *X*. In these tests, although bending was intentionally initiated at a variety of temperatures, the transition temperature was found to be quite constant, 113 ± 5 C. Generally, throughout this study the reproducibility of measured transition temperatures was found to be of the order of ±10 C at a level of 170 C and ±2 C at a level of 60 C.

It might be expected that at extremely rapid cooling rates, the temperature of the apparatus might lag that of the oil in which the apparatus is submerged. Since the recording thermocouple is immersed in the oil and is not in contact with the specimen or rotating drum erroneously low readings of fracture temperatures might occur. In order to determine whether such a difficulty existed, the transition temperatures of similar speci-

TABLE I.—EXAMPLES OF COOLING TIMES AND CORRESPONDING RATES AVAILABLE IN BEND TEST APPARATUS.

Temperature Range, deg Cent	Cooling Condition					
	Fast		Intermediate		Slow	
	Time, min	Rate, deg Cent per min	Time, min	Rate, deg Cent per min	Time, min	Rate, deg Cent per min
200 to 160	1.5	27	6	6.5	28	1.4
160 to 120	3.5	11	10	4.0	40	1.0
120 to 80	6	6.5	14.5	2.8	[a]	[a]
80 to 40	11.5	3.5	30	1.3	[a]	[a]

[a] Impractical because of the minimum strain rate presently available.

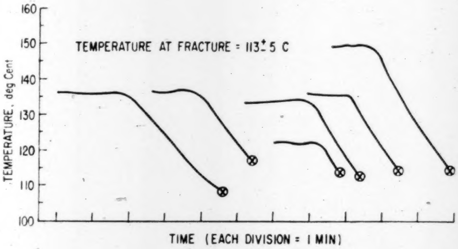


FIG. 4.—Several Temperature-Time Recorder Curves for an AgMg Compound on Which the Incidence of Fracture Is Denoted by an Encircled *X*.

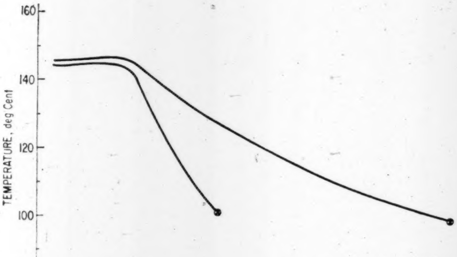


FIG. 5.—Temperature-Time Recorder Curves for Specimens of an AgMg Compound Cooled at a Fast and at a Slow Rate with the Incidence of Fracture Denoted by an Encircled *X*.

mens were measured at very different cooling rates; the curves for these experiments are shown in Fig. 5. The nearly identical transition temperatures measured permit the conclusion that, within the cooling rates attainable in the apparatus, any thermal difference between the specimen and bath is insignificant.

The diameter of the rotating drum presently dictates the length of specimen that can be wound around it and thus imposes a limit to the time and hence the temperature interval that can be covered in a single test at a given cooling rate. It was found, therefore, that the most efficient approach to determining the transition temperature of a material was to first approximate or bracket the transition temperature by interrupting the bending periodically while cooling was continued. After an approximate transition temperature is thus established, subsequent tests can be made with continuous bending during cooling.

APPLICATIONS

Previous studies of the influence of material variables on ductile-brittle transition temperature have shown effects of grain size (**3,4**), prestrain (**5,6**), purity (**7**), second phases (**8,9**), orientation (**10**), irradiation (**11**), alloying (**12**), and heat treatment (**2**).[3] For the purposes of assessing the reproducibility and limitations of the test method reported herein and of illustrating its application, some similar tests were made on wire samples of two materials—tungsten and the intermetallic compound AgMg.

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[3] The references cited are merely typical examples from a rather voluminous literature.

Effects of Strain Rate:

The effects of strain rate on transition temperature were studied in both tungsten and AgMg. Since the transition temperature of the commercial tungsten filament wire is quite low as a result of both prestrain and preferred orientation, it was necessary to pre-anneal the wire. A treatment of 2 hr in hydrogen at 1625 C was found to give transition temperatures well within the present range of the test equipment. The effects of strain rate on transition temperature for tungsten and AgMg are shown in Figs. 6 and 7 respectively. The reciprocal of the absolute transition temperature is seen to be linear with log strain rate as predicted by the theoretical models of Cottrell (13) and Petch (14). A summary of similar experimental results on the relation between strain rate and transition temperature has been provided by Magnusson and Baldwin (15).

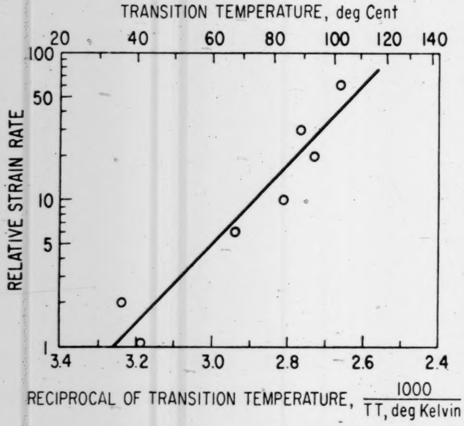

FIG. 6.—Relation of the Logarithm of the Relative Strain Rate to the Reciprocal of the Absolute Transition Temperature for Tungsten Wire Annealed 2 hr at 1625 C.

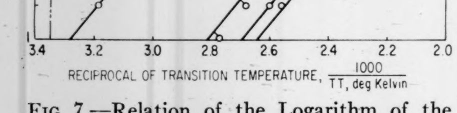

FIG. 7.—Relation of the Logarithm of the Relative Strain Rate to the Reciprocal of the Absolute Transition Temperature for Several Compositions of the Compound AgMg.

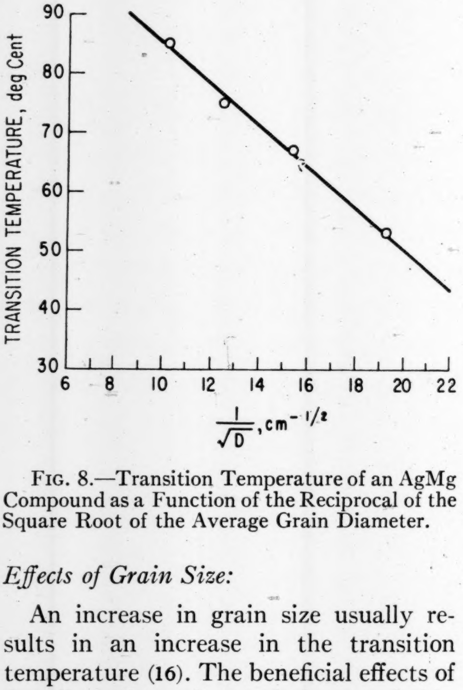

FIG. 8.—Transition Temperature of an AgMg Compound as a Function of the Reciprocal of the Square Root of the Average Grain Diameter.

Effects of Grain Size:

An increase in grain size usually results in an increase in the transition temperature (16). The beneficial effects of fine dispersions of second phase probably also act through grain refinement. The theoretical studies (13,14) show that the relationship to be expected is

$$T_c = kD^{-1/2} \qquad (1)$$

where T_c is the transition temperature and D the grain size. Both improved resistance to crack nucleation and improved resistance to crack propagation are thought to be responsible (17). AgMg wires were annealed in argon for 1 hr at a series of temperatures from 350 to 600 C with resulting grain sizes from 2.8×10^{-3} to 10.2×10^{-3} cm. Figure 8 shows that the results conform to Eq 1.

Effects of Heat Treatment:

In addition to its effect on grain size, heat treatment can alter the transition temperature of a material in several ways: precipitation, annealing out of prestrain or radiation damage, alteration of size of subgrain structure, change in the distribution of a segregating solute, removal of preferred orientations, and so on. An example of the effects of this parameter is afforded by annealing of the tungsten wire used in this study. Two-hour anneals over the temperature range from 1000 to 1625 C did not significantly alter the grain size but had a marked effect on the transition temperature as shown in Fig. 9. Although specific studies were not performed, it is believed that the observed increase in transition temperature results from loss of the benefits of the prestrain imposed by the commercial drawing process. Similar effects for AgMg have been noted by the authors (18). The effects on transition temperature of tungsten of annealing treatments over a wider and higher range of temperatures than those of Fig. 9 have recently been reported by Koo (19) who believes his observations are most likely due to changes in impurity distribution.

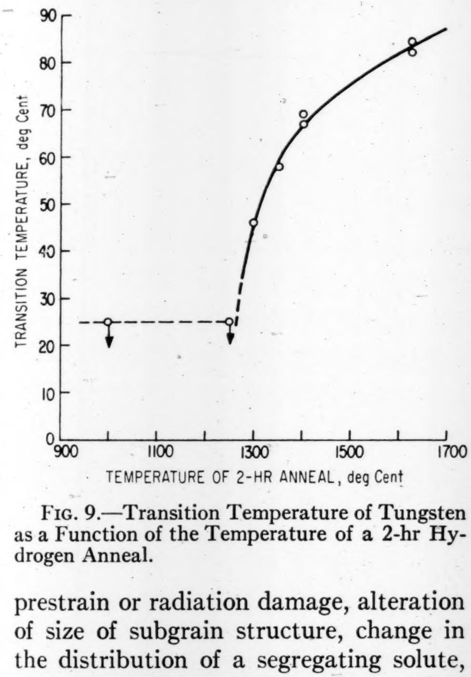

FIG. 9.—Transition Temperature of Tungsten as a Function of the Temperature of a 2-hr Hydrogen Anneal.

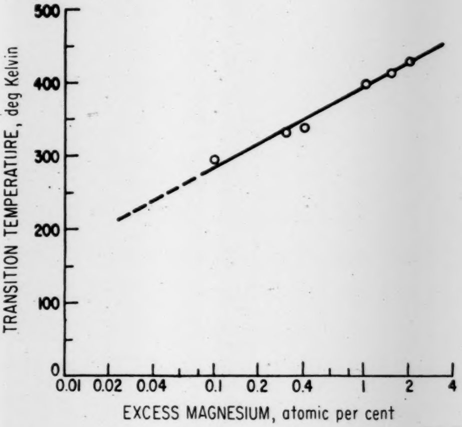

FIG. 10.—Absolute Transition Temperature of AgMg as a Function of the Logarithm of the Concentration of Magnesium in Excess of the Stoichiometric Composition, 50 Atomic per cent.

Effects of Composition:

An interesting effect of composition on transition temperature is afforded by AgMg. This compound exists over a broad range of solid solubility and may have either an excess of magnesium atoms or an excess of silver atoms relative to the 50 atomic per cent stoichiometric composition. The excess atoms have been shown to be accommodated as substitutional defects in the crystal structure (20) and to have important effects on mechanical properties (1,2). Figure 10 shows the linear variation of transition temperature with the logarithm of the concentration of excess magnesium. It is not known whether there is any fundamental significance to be attached to the functional relationship implied by this method of plot. However, the plot does illustrate, as has previously been noted for tensile flow (1), that minor deviations from stoichiometry have a much more potent effect than larger defect concentrations.

POSSIBLE FUTURE MODIFICATIONS AND EXTENSIONS OF THE EQUIPMENT

Modification of the equipment to accommodate other sizes and shapes of specimen as well as other ranges of strain rate is obvious and need not be detailed here.

In the tests herein reported, the incidence of fracture was observed directly and noted by hand on the recorder chart. The incorporation of a microswitch, an auxiliary recording pen, and appropriate circuiting to permit automatic recording of the moment of fracture is a straightforward improvement which could be made if desired.

Many materials of interest, for example, iron and the group V_A metals, will have subnormal transition temperatures even at the highest practicable strain rates. By substituting an appropriate cryostatic fluid, say 2 ethyl hexylchloride, for the silicone oil bath and circulating liquid nitrogen instead of tap water through the cooling coils, transition temperatures down to at least −130 C could be determined.

The apparatus might also be employed isothermally in various ways. By providing a drive which would supply a continuously accelerating rate of rotation of the drum, the critical strain rate for the ductile-brittle transition could be determined. With the substitution of a tapered mandrel for the cylindrical drum now used and the provision of an associated lateral feed, the critical strain to fracture could be measured. Appropriate compensation would be necessary to maintain constant strain rate.

SUMMARY

A new method of measuring transition temperatures is shown to give highly reproducible results. The convenience of establishing such a temperature with a single test on one specimen is demonstrated by a study of the effects of various metallurgical parameters on the transition temperatures of tungsten and the intermetallic AgMg. Various modifications of the equipment are suggested which would permit even broader applications.

Acknowledgments:

The authors are indebted to R. W. Kopp for assistance with the design of the equipment and to the U. S. Air Force for partial support under Contract No. AF 33(616)-7714.

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INFLUENCE OF SECTION SIZE ON MECHANICAL PROPERTIES AND FRACTURE TOUGHNESS OF 7075-T6 ALUMINUM, 6Al-6V-2Sn TITANIUM, AND AISI 4340 STEEL*†

By T. S. DeSisto,[1] F. L. Carr,[1] and F. R. Larson[1]

Synopsis

The influence of section size on mechanical properties—yield strength, tensile strength, elongation, reduction of area, and notched strength ratios—and fracture toughness was determined for 7075-T6 aluminum, 6Al-6V-2Sn titanium, and AISI 4340 steel. The tests were made on smooth-section tension specimens with diameters from 0.113 to 3.57 in., and sharply notched specimens with diameters from 0.160 to 5.0 in. The tensile fracture topography of the smooth and notched specimens is illustrated and discussed. The data show that yield and tensile strength are not changed appreciably by increasing section size, but ductility and toughness properties are affected.

Numerous investigations have been conducted at room temperature on smooth and notched round tension specimens. These investigations have been primarily concerned with the effect of section size on tensile properties (**1–3**)[2] and notch toughness (**4–6**). The recent interest in the fracture of metals and, in general, fracture mechanics has focused attention on the effect of section size on crack toughness of notched cylindrical specimens (**7,8**). This interest has also resulted in several recent publications of fracture surface analysis on both a microscopic scale (**9**) and a macroscopic scale (**10–12**). While previous work (**10,11**) was primarily concerned with the fracture topography of 4340 steel, the purpose of this study was to ascertain the fracture toughness of 4340 steel, 7075-T6 aluminum, and 6Al-6V-2Sn titanium over a broad range of sizes. Fracture topography measurements of 4340 steel as a function of section size augment prior investigations (**10,11**).

Test Materials and Procedures

Materials Chemical Analysis and Heat Treatment:

The materials used in this investigation together with their chemical analysis are shown in Table 1. The 7075-T6 aluminum and the 6Al-4V-2Sn titanium were tested as-received while the 4340 steel had the following heat treatment in the $5\frac{1}{4}$-in. section size:

Normalized at 1600 F for 4 hr, air cool.
Austenitized at 1550 F for 4 hr, oil quench.
Double tempered at 1000 F for 4 hr, oil quench.

Materials Testing Procedures:

Smooth and notched tension specimens of the sizes shown in Table 2 were tested. One specimen was tested for each size used. The smooth tension specimens were tested at 0.005 in. per in. per min to 0.2 per cent offset, then at 0.02 in. per min platen speed to fracture. The notched specimens were tested at a platen speed of 0.002 in. per min. Specimens 0.505 in. in diameter and smaller were tested in a 120,000-lb hydraulic testing machine, while the larger specimens were tested in a 2,400,000-lb hydraulic testing machine. In lieu of an extensometer for direct measurement of specimen strain on the smooth specimens 1.13 in. and larger, load platen displacement curves were obtained by use of a microformer deflectometer placed under the moving platen of the testing machine.

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* Presented at the Sixty-sixth Annual Meeting of the Society.

† The statements and opinions expressed in this article are those of the authors and do not necessarily indicate the views or policy of the U. S. Army Materiel Command.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

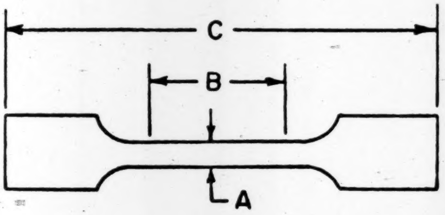
TABLE 1.—MATERIALS AND CHEMICAL COMPOSITIONS.

Material	Stock Diameter, in.	Chemical Analysis, weight per cent														
		Aluminum	Carbon	Chromium	Copper	Iron	Magnesium	Molybdenum	Manganese	Nickel	Phosphorus	Sulfur	Silicon	Tin	Vanadium	Zinc
7075-T6 Aluminum[a]	6¼	...	...	0.20	1.66	0.20	2.25	...	0.12	...	...	...	0.18	...	...	5.61
6Al-6V-2Sn Titanium[b]	4½	5.64	0.021	...	0.56	0.45	...	...	...	...	...	...	...	2.26	5.32	...
AISI 4340 Steel	5¼	...	0.451	0.82	...	...	...	0.24	0.73	1.90	0.005	0.010	0.35	...	...	...

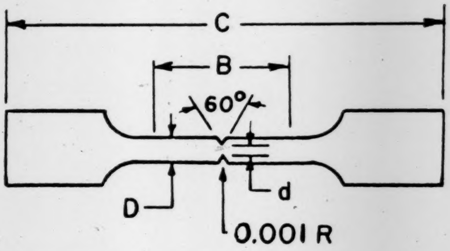
[a] Titanium, 0.01.

[b] Oxygen, 0.172; hydrogen, 0.0156; nitrogen, 0.007.

TABLE 2.—DIMENSIONS OF TEST SPECIMENS, IN.



A	B	C
0.113	0.4	1⅝
0.160	0.64	2¼
0.252	1.0	3
0.357	1.4	3½
0.505	2.0	4½
1.13	4.0	16¼
1.60	6.4	20
2.52	10.0	24
3.57	14.0	28



B	C	d	D
0.64	2¼	0.113	0.160
1.00	3	0.160	0.225
1.4	3½	0.252	0.357
2.0	4½	0.357	0.505
2.75	16¼	0.505	0.714
6.4	20	1.130	1.600
10.0	24	1.600	2.250
14.0	28	2.520	3.570
20.0	34	3.570	5.050

Fracture Topography Measurements:

The fractured surfaces of the 4340 steel specimens were examined at 10× and 20×, and the various zones of fracture configurations were measured with a stereoscopic microscope and a precision measuring engine which served as a stage. The measurement of each zone (fibrous, radial, and shear lip) (10) was

TABLE 3.—SMOOTH TENSILE PROPERTIES.

Material	Diameter, in.	Area, sq in.	Yield Strength, 0.2 per cent Offset, ksi	Tensile Strength, ksi	Per Cent Elongation	Reduction of Area, per cent
7075-T6 Aluminum	0.113	0.01	64.2	76.8	13.7	15.8
	0.160	0.02	61.5	74.75	12.5	15.1
	0.252	0.05	56.2	69.0	12.0	19.8
	0.357	0.1	61.5	75.2	16.4	21.6
	0.505	0.2	63.4	75.7	12.0	20.5
	1.13	1.0	60.0	72.5	...	17.7
	...	...	...	...	...	...
	2.52	5.0	59.2	72.4	10.0	14.7
	3.57	10.0	61.0	74.2	10.7	12.4
6Al-6V-2Sn Titanium	0.113	0.01	142.1	157.4	20.0	40.0
	0.160	0.02	130.0	146.5	19.1	44.4
	0.252	0.05	135.2	145.6	20.0	44.0
	0.357	0.1	140.2	148.5	18.6	43.2
	0.505	0.2	142.0	147.0	17.0	42.7
	1.13	1.0	126.0	138.5	14.4	33.5
	1.60	2.0	135.0	149.0	16.4	25.2
	2.52	5.0	...	142.6	10.0	31.4
AISI 4340 Steel	0.113	0.01	128.6	154.2	18.8	55.2
	0.160	0.02	128.4	155.3	15.0	48.4
	0.252	0.05	132.9	156.9	16.0	52.0
	0.357	0.1	128.0	152.8	17.9	52.1
	0.505	0.2	129.8	151.0	16.0	52.8
	1.60	1.0	130.0	147.0	8.1	47.2
	2.52	5.0	124.0	153.5	12.5	47.0

TABLE 4.—NOTCHED TENSILE PROPERTIES.

Material	Gross Diameter, in.	Notched Tensile Strength, ksi	Notched: Unnotched Tensile Strength Ratio	Notched: 0.2 Per Cent Yield Strength Ratio	K_{Ic}	K_t (Neuber)
7075-T6 Aluminum	0.160	92.0	1.25	1.51	15.2	10.7
	0.225	101.25	1.37	1.66	19.6	12.4
	0.357	89.5	1.21	1.47	22.2	15.5
	0.505	89.8	1.21	1.47	26.4	18.3
	0.714	88.0	1.19	1.44	30.8	21.5
	1.60	70.0	0.95	1.15	36.8	31.6
	2.25	55.0	0.745	0.90	34.2	37.0
	3.57	42.5	0.57	0.69	33.3	48.8
	5.05	58.3	0.79	0.96	54.2	55.4
6Al-6V-2Sn Titanium	0.160	225.8	1.53	1.64	37.4	10.7
	0.225	220.0	1.50	1.60	42.5	12.4
	0.357	210.0	1.43	1.53	52.0	15.5
	0.505	190.0	1.29	1.38	55.8	18.3
	0.714	179.0	1.22	1.30	62.6	21.5
	1.6	150.0	1.02	1.09	79.0	31.6
	2.25	123.5	0.84	0.90	77.0	37.0
	3.57	101.7	0.69	0.74	79.6	48.8
AISI 4340 Steel	0.160	229.5	1.50	1.78	38.0	10.7
	0.225	242.5	1.58	1.88	46.7	12.4
	0.357	227.0	1.48	1.76	56.3	15.5
	0.505	209.8	1.37	1.63	61.6	18.3
	0.714	218.0	1.42	1.69	76.3	21.5
	1.60	198.5	1.3	1.54	104.5	31.6
	2.25	206.0	1.35	1.60	128.0	37.0
	3.57	179.5	1.17	1.39	141.0	48.8
	5.05	176.0	1.15	1.37	163.5	55.4

determined in terms of the radius or the lineal length along the radius of the tensile fracture. Fracture zone measurements were made on diameters at 90-deg intervals on all specimens.

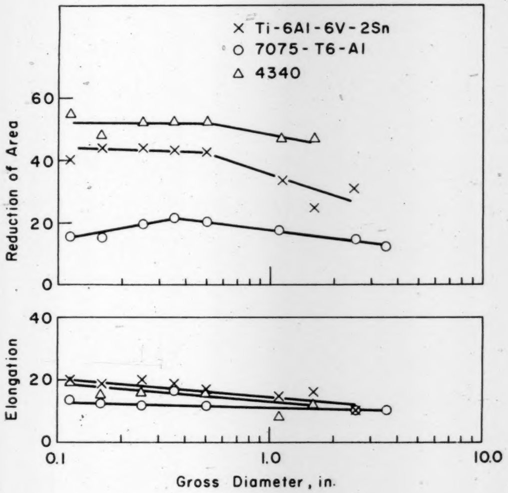


FIG. 1.—Reduction of Area and Elongation Versus Section Size.

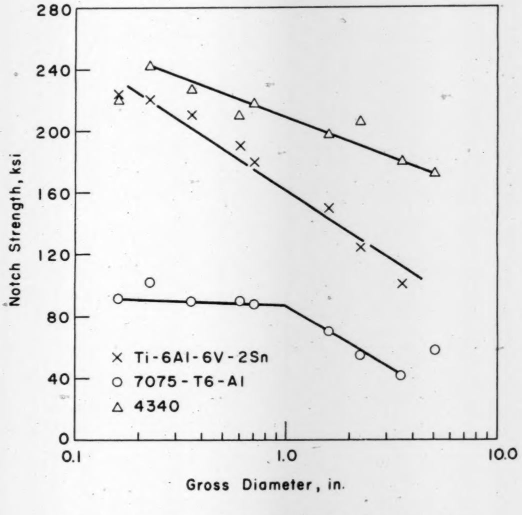


FIG. 2.—Notch Strength Versus Section Size.

TEST RESULTS

Mechanical Properties:

The smooth and notched tensile properties of 7075-T6 aluminum, 6Al-6V-2Sn titanium, and 4340 steel are tabulated in Tables 3 and 4 and plotted in Figs. 1 through 5. The yield and tensile strengths of the material tested did not show any significant changes with bar diameter. The ductility, however, plotted in Fig. 1, generally decreased with increasing bar diameter, with the reduction of area values for specimens larger than 0.505 in. decreasing more abruptly than the elongation values.

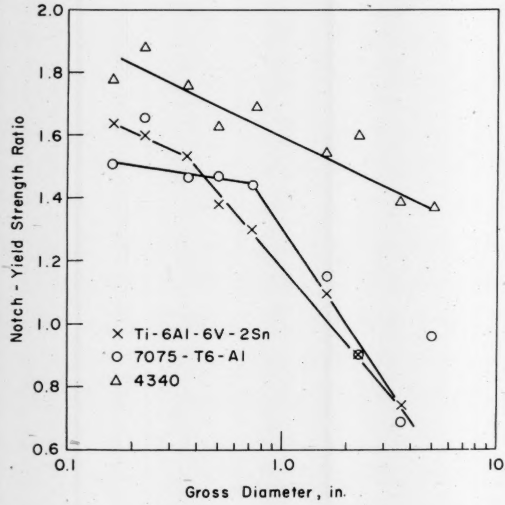


FIG. 3.—Notch-Yield Strength Ratio Versus Section Size.

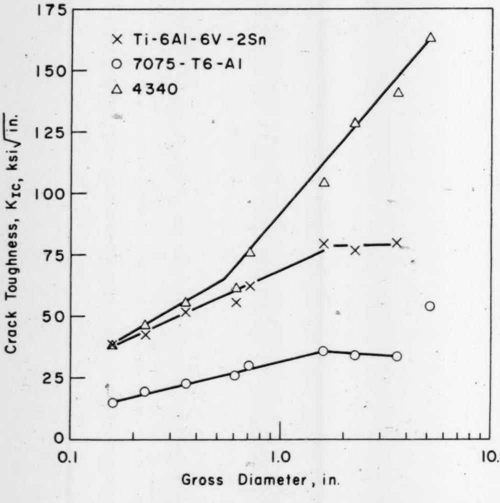
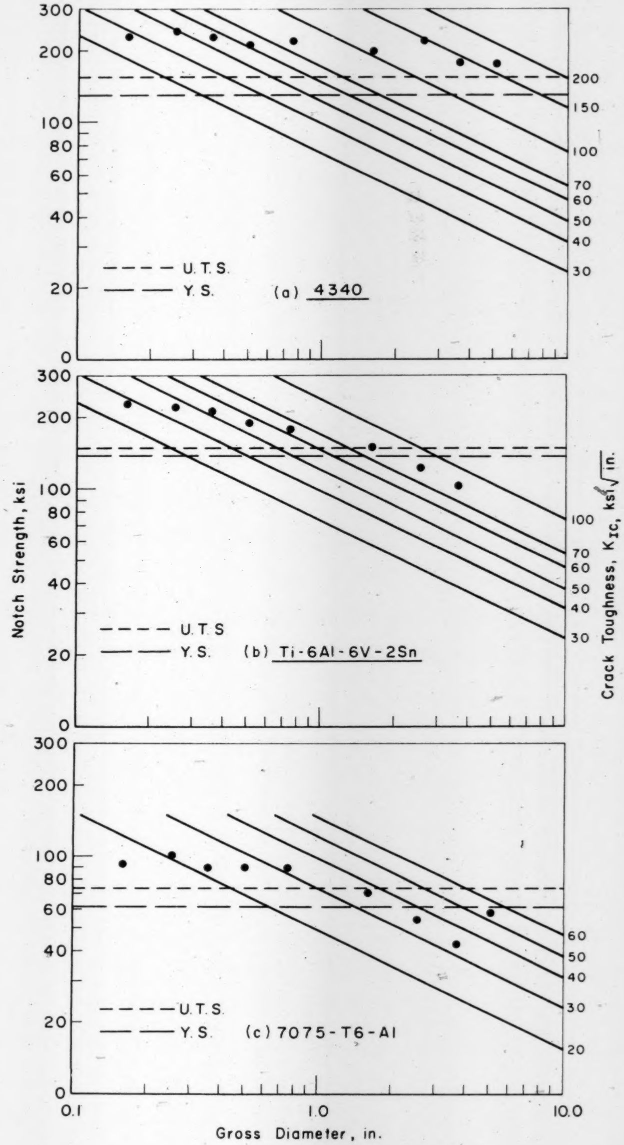


FIG. 4.—Crack Toughness, K_{Ic}, Versus Section Size.

The notch strength of the 4340 steel plotted in Fig. 2 decreases with increased bar diameter, reflecting size effects and increasing stress concentration, together with decreasing stress gradient. The decrease in notch strength of the 4340 steel tempered at 1000 F does not agree with recently published data by Klier et al (12) which show no change in notch strength with increasing section size from 0.3 in. to 1.5 in. The probable cause of this difference is the test section length of the Klier specimens which were not



(*a*) AISI 4340 steel. (*b*) 6Al-6V-2Sn titanium. (*c*) 7075-T6 aluminum.

FIG. 5.—Notch Strength Versus Section Size with K_{Ic} as the Parameter.

changed proportionally with increased section size (1.00 in. for the 0.3-in. diameter specimens and 1.5 in. for the 1.5-in. diameter specimens). The notch strength of the 7075-T6 aluminum generally remained constant with increased bar diameter to 0.505 in. Beyond this size, the notch strength decreased markedly to a low value of 42 ksi at the 3.57-in. size. The notch strength of the

5.05-in. specimen, however, was approximately 16 ksi higher than the 3.57-in. specimen. This increase in notch strength could not be solved by a hardness survey. The notch strength of the 6Al-6V-2Sn titanium alloy decreased from 224,000 ksi for the 0.160-in. specimen to 100,000 ksi for the 3.57-in. specimen. The notch: yield strength ratios plotted in Fig. 3 generally follow the same trend as the notch strength plotted in Fig. 2. Notch: yield strength ratios below 1.0 were obtained with 7075-T6 aluminum and 6Al-6V-2Sn titanium specimens larger than 1.60-in. diameter.

TABLE 5.—LINEAL ZONE SIZE.

Material	Smooth Zone Size, in.				Notched Zone Size, in.		
	Specimen Diameter	Fibrous	Radial	Shear Lip	Shank Diameter	Fibrous	Radial
AISI 4340 Steel......	3.57	...	...	...	5.05	0.023	1.761
	2.52	0.056	0.727	0.123	3.57	0.020	1.231
	1.60	0.059	0.445	0.081	2.25	0.040	0.756
	1.13	...	...	...	1.60	0.022	0.542
	0.505	0.015	0.131	0.031	0.714	0.029	0.221
	0.357	0.012	0.086	0.029	0.505	0.034	0.139
	0.252	0.015	0.050	0.023	0.357	0.022	0.101
	0.160	0.015	0.021	0.017	0.225	0.015	0.061
	0.113	0.005	0.019	0.013	0.160	0.022	0.032
6Al-6V-2Sn Titanium	2.52	...	...	0.177			
	1.60	...	...	0.136			
	1.13	...	...	0.099			
	0.505	...	...	0.045			
	0.357	...	...	0.032			
	0.252	...	...	0.024			
	0.160	...	...	0.016			
	0.113	...	...	0.018			
7075-T6 Aluminum..	3.57	...	...	0.242			
	2.52	...	...	0.154			
	1.60	...	...	...			
	1.13	...	...	0.098			
	0.505	...	...	0.123			
	0.357	...	...	0.065			
	0.252	...	...	0.028			
	0.160	...	...	0.014			
	0.113	...	...	0.044			

It has been suggested (7) that cylindrical sharply notched specimens can be used to determine plane section fracture toughness, K_{Ic}, properties of materials. This technique is suitable if the material being evaluated is brittle. In the case where materials may be moderately tough, it is necessary to test fairly large specimens. This is illustrated for the crack toughness data of 4340 steel shown in Fig. 4, where even at the 5.05-in. specimen diameter the notch:yield strength ratio was 1.37, considerably higher than the 1.10 required for valid K_{Ic} determinations. On the other hand, the lower toughness 7075-T6 aluminum and 6Al-6V-2Sn titanium meet the requirements at the 1.60-in. section size and K_{Ic} values of 36.8 ksi$\sqrt{\text{in.}}$ and 79 ksi$\sqrt{\text{in.}}$ are obtained for the aluminum and titanium alloys. A review of Fig. 3 will show that notch:yield strength ratios 1.09 and 1.15 were obtained for the aluminum and titanium at the 1.60-in. section size. Wundt-type summary plots of the notch strength of 7075-T6 aluminum, 6Al-6V-2Sn titanium, and 4340 steel as a function of section size are shown in Figs. 5(*a*), (*b*), and (*c*) with K_{Ic} as the parameter. Yield and tensile data are also included in the plots.

Fracture Topography:

Fracture of the smooth specimens of 4340 steel initiated at an inclusion or void and propagated toward the periphery. This process resulted in three zones of configurations which progressed from the origin in the following sequence: (1) fibrous zone composed of circumferential ridges indicative of slow crack growth; (2) radial zone comprised of radial markings which appear to be shear elements indicative of accelerated crack propagation; and (3) shear lip zone containing a smooth shear element near the termination of the fracture.

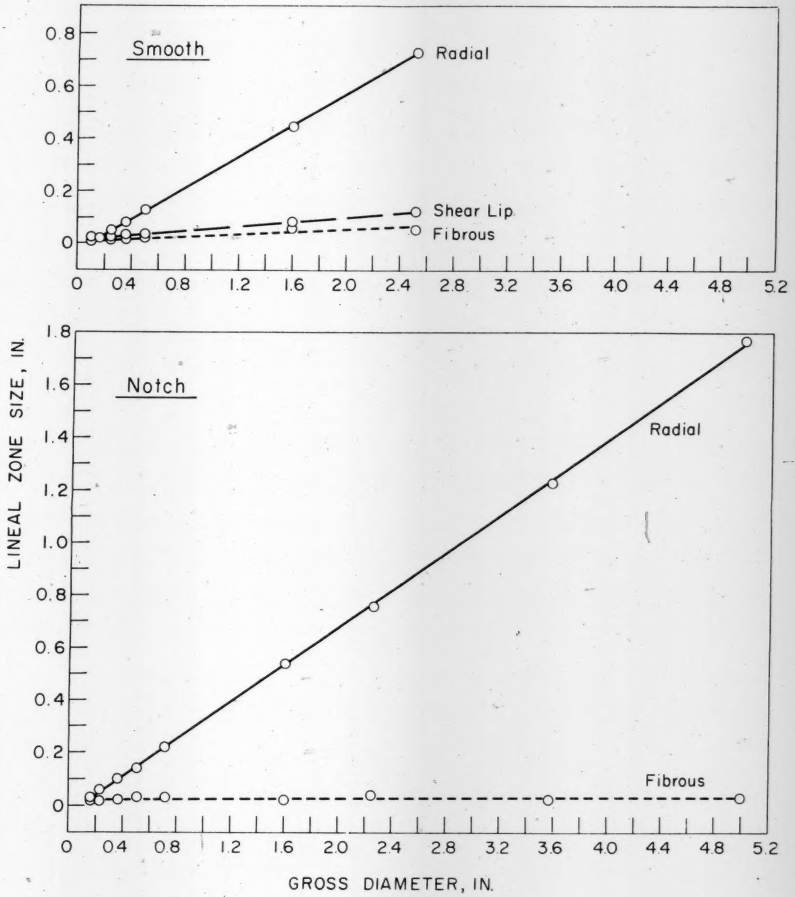


Fig. 6.—Lineal Zone Size Versus Section Size of AISI 4340 Steel.

Measurements of these zones on the fractured specimens indicated that the size of each zone increased with increasing section size, but the rate of increase for each respective zone differed. The various zone sizes are tabulated in Table 5 and illustrated as a function of section size in Fig. 6. It is noted that both the fibrous zone and the shear lip zone increased with increasing bar diameter. It is also noted that the fibrous zone is approximately one-half the shear lip width. It is worth while mentioning, however, that the increase in fibrous zone is actually a decrease in per cent fibrous fracture of the fracture surface. The increase in shear lip size from 0.013 in. for the 0.113-in. specimen to 0.123 in. for the 2.52-in. specimen does not agree with recently published data (**13**) which show no change in shear lip size of specimens

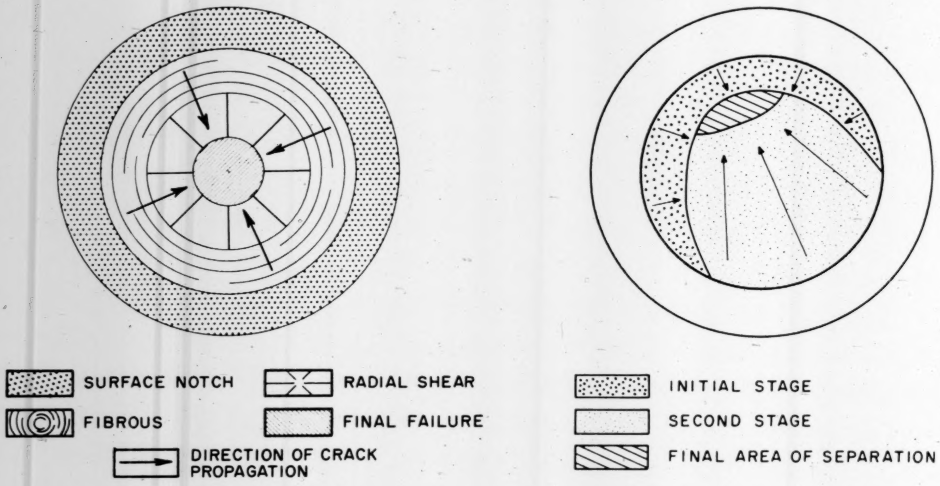

SURFACE NOTCH
RADIAL SHEAR
FIBROUS
FINAL FAILURE
DIRECTION OF CRACK PROPAGATION
INITIAL STAGE
SECOND STAGE
FINAL AREA OF SEPARATION

FIG. 7.—(*a*) Fracture Surface Markings in Notch Round Specimens of AISI 4340 Steel and (*b*) Various Fracture Stages of Notched Specimens of 7075-T6 Aluminum and 6Al-6V-2Sn Titanium.

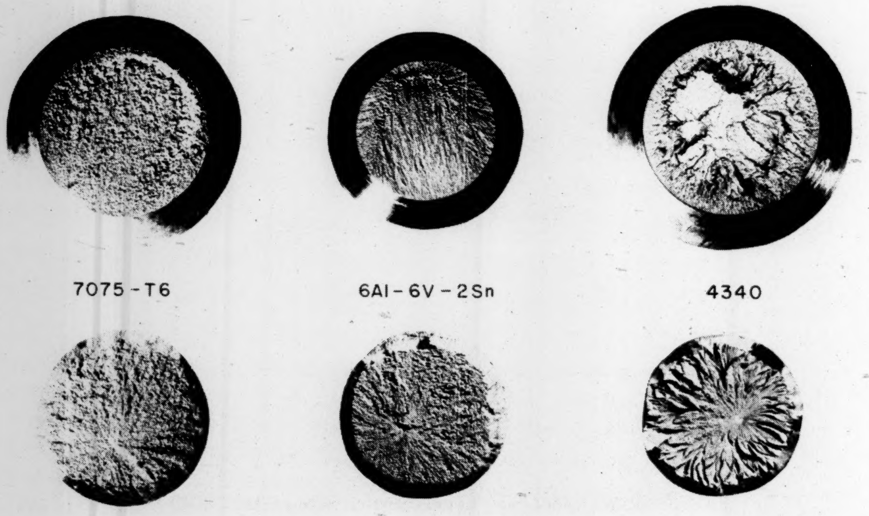

7075-T6
6Al-6V-2Sn
4340

FIG. 8.—Tensile Fracture Topography of Largest Smooth and Notched Specimens Tested.

of 4340 steel from 0.357-in. diameter to 3.57-in. diameter.

Crack initiation in the notched bar of 4340 steel occurred at the notch and progressed circumferentially toward the interior of the specimen. Although the fibrous and radial zones of the notch specimens are in opposite locations to those of the smooth specimens, the sequence in both cases is identical with respect to the direction of crack propagation. These characteristics are illustrated in Fig. 7(*a*). The fibrous zone of the notch specimen generally remained constant with increasing section size, increasing stress concentration factor, and decreasing stress gradient reflecting the maximum slow crack growth possible for this material condition. The texture of the radial zone of the fracture surfaces of both notch and smooth specimens is rougher at the area of final separation reflecting an increased rate of crack propagation (**14**). Evidence of this is shown in Fig. 8, a composite photograph of the fractured surfaces of the largest smooth and notched specimens used in this investigation.

The internal final area of separation of the notched specimens, as evidenced by this investigation, does not agree with the description advanced by Klier and Weiss (**12**). The assumption advanced by these authors suggested the area of rough texture to be a second nucleus and that propagation is in a direction opposite to that inferred by the interpretation of this study. Since any concept of crack propagation must at present be based on vestigial evidence, individual interpretations may be somewhat controversial.

Discrimination of the fibrous and radial zones, previously described for the steel specimens, was difficult for the fractured titanium specimens; therefore only the shear lip measurements are included in Table 5. The shear lip increased in size with increasing specimen diameter. The fracture topography of the notched specimens of this alloy can be described as three stages illustrated in Fig. 7. The initial stage extended completely around the circumference of the fractured surface on both the 0.160-in. and 0.357-in. diameter specimens. Thus for the smaller specimens the initial stage appeared as a ring, but on the larger specimen this stage was crescent or "horseshoe" in shape. The second stage (Fig. 7(*b*)), which was more rapid in propagation, started as an arc at a location approximately 180 deg from the origin and advanced through the specimen, converging toward the origin until the final area of separation was reached. This process resulted in the second stage having the general shape of a fan. The direction of propagation can be ascertained by following the increasing roughness of the markings in this second stage.

The fracture topography of the 7075-T6 aluminum alloy was similar to that described for the titanium alloy. Measurements determined that the shear lip also increased in size with increasing specimen diameter. The first stage of fracture of the notched specimens of this alloy also appeared as a ring, and extended completely around the circumference of the fractured surface at specimen diameters less than 1.60 in. For larger sizes this stage receded circumferentially and acquired the shape of a horseshoe, but never attained the crescent shape observed on the fractured specimens of 6Al-6V-2Sn titanium.

Summary

Mechanical properties of 7075-T6 aluminum, 6Al-6V-2Sn titanium, and 4340 steel have been determined on smooth specimens from 0.113-in. diameter to 3.57-in. diameter and on notched specimens from 0.160-in. diameter to 5.05-in. diameter. Crack toughness values and some fracture topography measurements were also obtained.

The yield and tensile strengths of the materials tested did not show any significant changes with bar diameter. The notch strength and notch:yield strength ratios of all materials decreased with increasing bar diameter. Notch:yield strength ratios below 1.0 were obtained with specimens of 7075-T6 aluminum and 6Al-6V-2Sn titanium larger than 1.60 in. in diameter. The crack toughness, K_{Ic}, of 4340 steel could not be determined as there was considerable plastic yielding even at the 5.05-in. size. However, K_{Ic} values of 36.8 ksi$\sqrt{\text{in.}}$ and 79 ksi$\sqrt{\text{in.}}$ were obtained for the 7075-T6 aluminum and 6Al-6V-2Sn titanium alloys at the 1.60-in. specimen diameter.

Fracture of the smooth specimens initiated in the interior of the specimens and propagated toward the periphery, while fracture of the notched specimens initiated at the base of the notch and propagated toward the interior of the specimen.

Fracture zone measurements of the smooth specimens showed that the shear lip size increased with specimen diameter for all materials tested. The fibrous zone of the 4340 specimens also increased with specimen diameter.

The fibrous zone of the notched 4340 specimens did not change appreciably with increasing specimen diameter, reflecting the maximum slow crack growth for this material condition.

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DISCUSSION

MR. SAMUEL EPSTEIN.[1]—Was there leveling off in the decrease in toughness with increase in section size after a certain section size was reached?

MR. T.S. DESISTO (*author*).—At notch yield ratios of 1.1 or less, there is no change in plane strain fracture toughness with increased specimen size.

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COMPARISON OF TEST SPECIMENS FOR NOTCH SENSITIVITY AND FRACTURE TOUGHNESS OF HIGH-STRENGTH SHEET MATERIALS*

By Charles F. Hickey, Jr.,[1] and Frank R. Larson[1]

Synopsis

A variety of test specimens and test methods are currently being used by several investigators to evaluate materials for space applications. In general these tests are being conducted on high-strength sheet materials using specimens containing notches of one sort or another. The purpose of this program was to compare various test specimens for determination of notch sensitivity and fracture toughness.

Specimens considered were: (1) 1-in. wide edge notch, (2) 1.5-in. wide center notch, and (3) 3-in. wide center notch. Notch sensitivity data were obtained from all specimens, whereas fracture toughness data, based upon the ink stain and per cent shear method, were gathered primarily from the 3-in. wide specimen. Materials considered include AMS 6434, H-11, 300M, and PH 15-7 molybdenum steels and 6Al-4V titanium. Two or more strength levels per alloy were investigated and tested temperatures ranged from +165 to −80 F.

Results of this investigation indicate that of the designs considered the 3-in. wide specimen generally offers the best test to measure notch sensitivity or fracture toughness.

With the increased use of high-strength sheet materials, it has become imperative that test methods be established that will provide either quantitative or qualitative information on brittle fracture. In 1959, the ASTM established the Committee on Fracture Testing of High-Strength Sheet Materials for this purpose; their recommendations have been published in the *ASTM Bulletin* (**1,2**).[2] The committee recommended three general types of specimens—an edge notch and two center notch designs. The edge notch specimen has a 1-in. gage width, a 15 per cent notch on each edge, and a machined notch radius of 0.001 in. or less. Such design has been commonly referred to as the NASA or Brown type (Fig. 1 (*a*)). A companion smooth tension specimen is tested and the data are reported in the form of sharp-notch tensile strength to smooth tensile or yield strength ratio. The two other designs recommended are center notch specimens having widths of 1.5 and 3 in. The notch length for both designs is approximately one-third of the specimen width. Such specimens are tested in either a fatigue cracked or sharp machined notched condition (0.001 in. or less). Net section stress is generally re-

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* The statements and opinions expressed in this article are those of the authors and do not necessarily indicate the views or policy of the U.S. Army Material Command.

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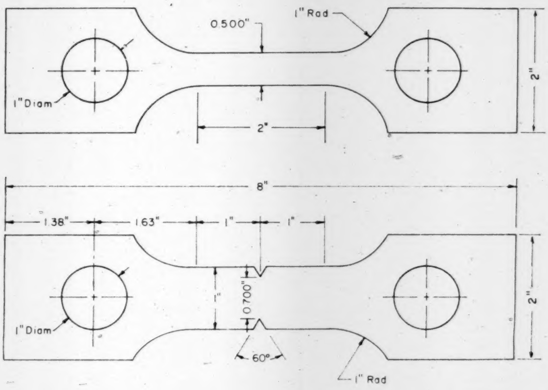
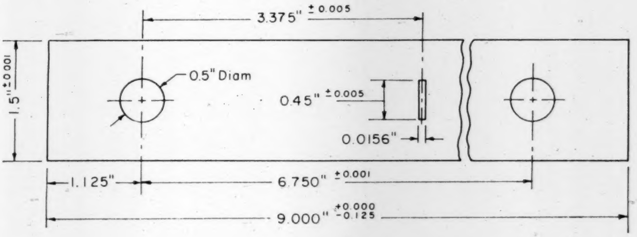
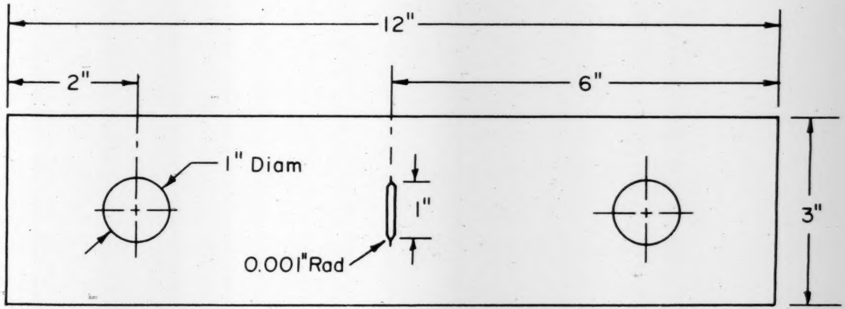
[2] The boldface numbers in parentheses refer to the list of references appended to this paper.


0.500"
1" Rad
1" Diam
2"
2"
8"
1.38"
1.63"
1"
1"
1"
0.700"
1" Diam
2"
60°
1" Rad

(*a*) NASA (Brown) type notched and unnotched tension specimens.


3.375" ± 0.005
1.5" ± 0.001
0.5" Diam
0.45" ± 0.005
0.0156"
1.125"
6.750" ± 0.001
9.000" +0.000 -0.125

(*b*) NRL (Srawley) type fracture toughness specimen.


12"
2"
6"
1" Diam
1"
3"
0.001" Rad

(*c*) NRL (Irwin-Kies) type fracture toughness specimen.

FIG. 1.—Tension and Fracture Toughness Specimens Used in Study.

ported on the 1.5-in. specimen, and fracture toughness data, K_c, are obtained from the 3-in. specimen. These specimens have been commonly referred to as the NRL (Srawley) and NRL (Irwin-Kies) type (Figs. 1 (*b*) and (*c*) respectively).

Other tests that have been used in obtaining notch sensitivity data for high-strength sheet materials include the Charpy, bulge, and bend tests (**3**). These have not been used as extensively as the aforementioned tests; nevertheless, valuable information has been obtained from them.

TABLE 1.—CHEMICAL COMPOSITION OF MATERIALS TESTED.

Alloy	Element, per cent										
	Carbon	Silicon	Sulfur	Phosphorus	Manganese	Nickel	Chromium	Vanadium	Molybdenum	Aluminum	Titanium
AMS 6434	0.38	0.40	0.04[a]	0.04[a]	0.68	1.87	0.77	0.25	0.38	...	...
H-11	0.41	0.89	0.006	0.012	0.33	0.10	5.15	0.54	1.24	...	...
300M	0.40	0.152	0.025[a]	0.025[a]	0.66	1.68	0.83	0.06	0.35	...	...
PH 15-7 Mo	0.08	0.34	0.04[a]	0.04[a]	0.46	7.20	14.90	...	2.09	1.00	...
Ti-6Al-4V	...	...	...	...	...	...	...	3.91	...	6.00	90.04

[a] Not determined; maximum allowable specification requirement.

TABLE 2.—HEAT TREATING PROCEDURES FOR INVESTIGATED MATERIALS.

Alloy	Yield Strength Level, ksi	Heat Treating Procedures
AMS 6434	210	Austenitize at 1600 F; oil quench; temper at 600 F for 2 hr
H-11	210	Preheat at 1450 F; soak at 1850 F for 20 min; air cool; double temper at 1000 F, 1 hr per temper
300M	210	Austenitize at 1575 F; oil quench; temper at 650 F for 1 hr
PH 15-7 Mo	210	Austenitize at 1750 F; air cool to room temperature; cool to −100 F, hold 8 hr; warm to room temperature; age at 950 F for 1 hr; air cool
AMS 6434	190	Austenitize at 1600 F; oil quench; temper at 875 F for 2 hr
H-11	190	Preheat at 1450 F; soak at 1850 F for 20 min; air cool; triple temper at 925 F, 3 hr per temper
300M	190	Austenitize at 1575 F; oil quench; temper at 900 F for 1 hr
PH 15-7 Mo	190	Austenitize at 1750 F; air cool to room temperature; cool to −100 F, hold 8 hr; warm to room temperature; age at 850 F for 1 hr; air cool
Ti-6Al-4V	165	Solution treat at 1700 F for 30 min; water quench; age at 900 F for 8 hr; air cool
	150	Solution treat at 1575 F for 30 min; water quench; age at 1000 F for 8 hr; air cool
	130	Solution treat at 1450 F for 2 hr; furnace cool; age at 1100 F for 8 hr; air cool

The purpose of this investigation was to compare notch tension test specimens for obtaining the notch sensitivity or fracture toughness of high-strength sheet material. Primary emphasis was placed on the above-mentioned edge notch and center notch type tension specimens. Materials considered included four high-strength steels and one titanium alloy. Two or more strength levels were investigated and the materials were tested at +165, +76, and −80 F.

The testing was conducted on this program at the U. S. Weapons Plant on Contract No. MIPR 19-066-60-4 under the supervision of the U. S. Army Materials Research Agency. The project was entitled "Correlation of Notch Sensitivity and Fracture Toughness Test Methods."

TEST MATERIALS AND PROCESSING

The materials selected for this investigation were AMS 6434, H-11, 300M, PH 15-7 molybdenum steels, and 6Al-4V titanium alloy. Sheet thicknesses ranged from 0.093 in. to 0.109 in. The chemical analyses of the five alloys are shown in Table 1.

The various steels were heat treated to 0.2 per cent yield strength levels of 190 and 210 ksi, while the 6Al-4V titanium alloy was heat treated to 0.2 per cent yield strength levels of 130, 150, and 165 ksi. Complete descriptions of the heat treating procedures are listed in Table 2 and the smooth tensile properties at room temperature in Table 3.

Both the 1- and 3-in. specimens were tested with machined notches which had a root radius of 0.001 in. or less. The 1.5-in. specimen design was notched by machining and then fatigue cracked to a depth of 0.010 to 0.015 in. at each edge of the notch, using tension-tension fatigue stresses.

Slow crack extension was measured only for the 3-in. specimen design employing the common crack indicators of India ink for tests at +76 and +165 F and aniline blue dissolved in methyl ethyl ketone for tests conducted at −80 F.

TABLE 3.—MECHANICAL PROPERTIES OF INVESTIGATED MATERIAL.

	Yield Strength Level, ksi	Yield Strength at 0.2 per cent Offset, ksi	Ultimate Tensile Strength, ksi	Elongation in 2 in., per cent	Reduction of Area, per cent
AMS 6434	210	208	235	6.5	35.0
H-11	210	207	277	8.5	32.4
300M	210	211	260	7.0	15.2
PH 15-7 Mo	210	211	232	7.0	22.0
AMS 6434	190	193	205	8.0	35.0
H-11	190	194	295	9.0	28.7
300M	190	190	208	9.5	36.8
PH 15-6 Mo	190	189	217	7.0	25.5
Ti-6Al-4V	165	165	181	6.5	19.0
	150	149	154	10.5	36.2
	130	130	134	12.0	33.0

PROCEDURE AND TEST RESULTS

The tension testing was carried out on a 120,000-lb Baldwin-Southwark universal hydraulic testing machine using a crosshead speed of 0.35 in. per min. Specimens were tested at three temperatures, +165, +76 (room temperature), and −80 F, in order to cover the normal transition range of ductile-to-brittle behavior for these alloys and heat treatments. As mentioned previously, the main emphasis of this program was the comparison of the three specimen geometries recommended by the ASTM fracture committee (1,2). The three types—1-in. edge notch, 1.5-in. center notch, and 3-in. center notch—are shown in Figs. 1 (*a*) to (*c*), respectively.

The results of this testing procedure are tabulated in Table 4. Characteristic of each specimen design is the measurement of certain values from which a relative indication of the notch sensitivity or fracture toughness is obtained. Although different procedures are used for each specimen, it will be shown later that the information can be related through fracture mechanics in most cases. The specific data usually reported for the various specimen designs as listed in Table 4 are as follows:

1. One-inch wide edge notch (*NASA or Brown type*).—This specimen employs a ratio of notched to smooth strength as a measure of toughness so

TABLE 4.—NOTCH SENSITIVITY AND FRACTURE TOUGHNESS DATA.

NOTE.—All values are the average of three tests.

				1-in. Specimen				1.5-in. Specimen				3-in. Specimen						
Alloy	Sheet Thickness, in.	Yield Strength (0.2 per cent) Level, σ_y, ksi	Test Temperature, deg F	Ultimate Tensile Strength, σ_u, ksi	Notch Strength, σ_N, ksi	Notch Yield Strength Ratio, σ_N/σ_Y	Notch Tensile Strength Ratio, σ_N/σ_u	Gross Stress, σ_G, ksi	Net Section Stress, σ_n, ksi	Shear, per cent	Fracture Toughness, K_{c3}, ksi $\sqrt{\text{in.}}$	Gross Stress, σ_G, ksi	Notch Strength, σ_N, ksi	Nominal Stress, σ_n, ksi	Slow Crack Extension, $2a$, in.	Shear, per cent	Fracture Toughness,[a] ksi $\sqrt{\text{in.}}$ K_{c3}	Fracture Toughness,[a] ksi $\sqrt{\text{in.}}$ K_{c1}
AMS 6434	0.093	210	+165	247	185	0.88	0.75	117	179	>95	171	102	153	208	1.56	>95	204	231
			+76	246	188	0.89	0.76	119	195	>95	175	109	164	228	1.56	>95	221	274
			−80	256	194	0.92	0.76	80	122	30	91	59	89	103	1.27	40	103	89
H11	0.100	210	+165	264	124	0.59	0.47	40	62	<5	40	55	83	89	1.21	0 to 5	90	83
			+76	273	122	0.58	0.45	39	59	<5	39	52	78	80	1.04	5	85	72
			−80	283	99	0.47	0.35	27	43	<5	27	41	62	69	1.23	0	66	62
300M	0.109	210	+165	267	194	0.92	0.73	127	199	>95	196	95	143	200	1.58	>95	191	206
			+76	260	200	0.95	0.77	122	190	>95	185	107	161	229	1.60	>95	219	196[c]
			−80	245	185	0.88	0.76	71	107	20	77	54	81	88	1.18	20	90	81
PH 15-7 Mo	0.100	210	+165	222	147	0.70	0.66	66	107	20	71	54	81	85	1.12	5 to 10	89	77
			+76	230	133	0.63	0.58	63	97	<5	64	43	65	67	1.07	0 to 5	69	60
			−80	250	106	0.50	0.42	33	50	<5	33	31	47	48	1.05	0 to 5	50	43
AMS 6434	0.093	190	+165	197	174	0.92	0.88	117	184	>95	177	108	162	214	1.49	>95	218	237
			+76	200	195	1.03	0.97	117	180	>95	177	108	162	208	1.45	>95	218	228
			−80	212	174	0.92	0.82	120	187	>95	185	111	167	212	1.42	<95	225	225
H11	0.100	190	+165	271	151	0.79	0.56	40	62	<5	40	51	77	85	1.18	0 to 5	84	77
			+76	280	147	0.77	0.53	33	51	<5	33	50	75	78	1.08	5	82	71
			−80	289	118	0.62	0.41	27	41	<5	27	33	50	58	1.27	0 to 5	53	83
300M	0.109	190	+165	207	187	0.98	0.90	124	196	>95	197	98	147	190	1.46	>95	197	195
			+76	219	197	1.04	0.90	121	188	>95	190	102	158	213	1.56	>95	207	230
			−80	220	176	0.93	0.80	75	113	20	82	60	96	110	1.24	15	100	94
			+165	225	124	0.65	0.55	94	150	60	122	82	123	129	1.09	5 to 10	139	121

PH 15-7 Mo...	0.100	190	+76	230	132	0.69	0.57	93	143	25	106	52	78	81	1.06	0 to 5	85	73
			−80	248	89	0.47	0.36	39	62	<5	39	34	51	55	1.10	25	57	48
Ti-6Al-4V.....	0.093	165	+165	170	140	0.85	0.82	69	104	80 to <5	97	51	77	102	1.49	30 to 35	88	93
			+76	178	130	0.79	0.73	53	82	80 to 85	67	48	72	95	1.41	30 to 35	83	83
			−80	201	108	0.65	0.54	45	69	20 to 40	52	44	66	76	1.28	25 to 30	70	70
		150	+165	147	163	1.09	1.10	100	153	60 to 75	146	84	126	237	1.90	75 to 80	166	182[c]
			+76	156	162	1.08	1.04	[b]	[b]	...	[b]	79	119	184	1.68	45 to 80	155	153[c]
			−80	175	162	1.08	0.93	86	140	60	113	63	95	136	1.59	35 to 40	113	130
		130	+165	125	146	1.12	1.17	84	129	50 to 85	125	156	116	191	1.74	40 to 80	154	156[c]
			+76	135	155	1.19	1.16	[b]	[b]	...	[b]	178	120	227	1.95	75 to 80	163	178[c]
			−80	155	171	1.32	1.01	101	154	75	162	[b]	[b]	[b]	[b]	[b]	[b]	[b]

[a] $K_{c1} = \sigma\sqrt{Wq_1}$; $K_{c3} = \sigma\sqrt{Wq_3}$.

[b] No reliable data obtained; specimens failed in both pinhole and templin grips.

[c] $K_{c1} = \sigma\sqrt{W \tan \pi a/W}$.

that a companion smooth specimen must be tested along with the notch specimen.

(*a*) σ_N = Notch strength: maximum load divided by the initial area under the notch.

(*b*) σ_u = Ultimate tensile strength (smooth specimen).

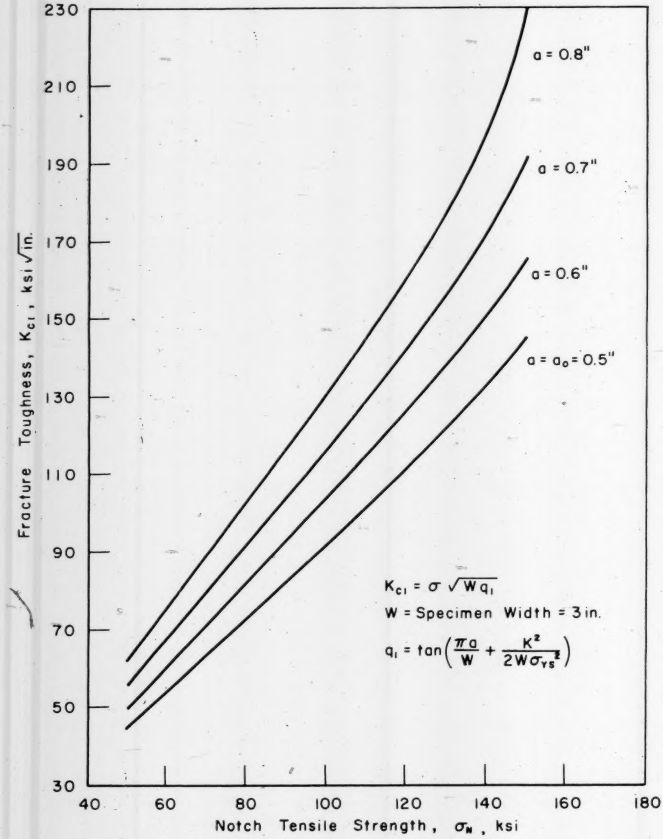


FIG. 2.—Notch Tensile Strength Versus Fracture Toughness as a Function of Slow Crack Extension.

(*c*) σ_y = Tensile yield strength (smooth specimen).

(*d*) σ_N/σ_u = Notch tensile strength ratio.

(*e*) σ_N/σ_y = Notch yield strength ratio.

2. *One and one-half-inch wide center notch fatigue cracked* (*NRL, Srawley type*).—This specimen type is usually used to obtain fracture appearance and net section stress transitional data over a range of test temperatures.

(*a*) σ_N = Net fracture stress: maximum load divided by initial supporting cross-section areas (same as 1(*a*) notch strength) but refers to center notch specimens.

(*b*) σ_n = Net fracture stress (nominal stress) has also been determined by dividing maximum load by the area remaining at the onset of rapid fracture, usually measured by ink stain method.

(*c*) a = Slow crack extension measured by ink stain.

(*d*) P = Per cent shear which is a percentage of oblique fracture to total fracture surface.

3. *Three-inch wide center notch specimen* (*NRL, Irwin-Kies type*).—This type is recommended for fracture mechanics data.

(*a*) σ = Gross fracture strength: maximum load divided by the area, based upon entire specimen width.

(*b*) σ_N = Notch strength same as 1(*a*).
(*c*) σ_n = Net fracture stress (nominal stress) as in 2(*b*).
(*d*) a = Slow crack extension as in 2(*c*).
(*e*) P = Per cent shear as in 2(*d*).
(*f*) σ_y = Yield strength of smooth specimen as in 1(*c*).
(*g*) K_{c1} = Fracture toughness, ink stain method, calculated as in reference (**1**).
(*h*) K_{c3} = Fracture toughness per cent shear method, also calculated as in reference (**1**).

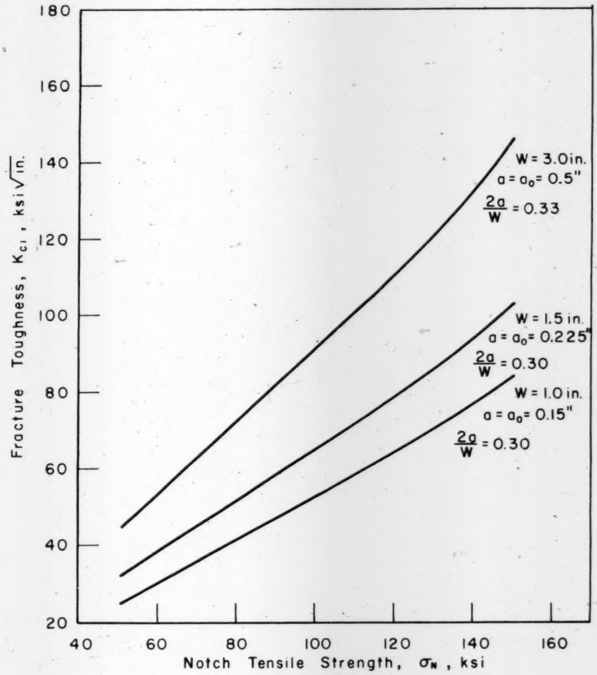


FIG. 3.—Notch Tensile Strength Versus Fracture Toughness as a Function of Specimen Width.

COMPARISON OF TEST SPECIMENS

Since it is clear, from fracture mechanics, that the notch strength and K_c are uniquely related for one specimen size provided that general yielding has not occurred, it should be possible to compare various specimen sizes based upon the notch strength-K_c interrelationships.

The fracture toughness, K_c, is related to gross fracture strength from the general equation, as follows:

$$K_c = \sigma\sqrt{wq} \quad \text{.............} (1)$$

where

$$q = \tan\left(\frac{\pi a}{w} + \frac{K^2}{2w\sigma_y{}^2}\right)$$

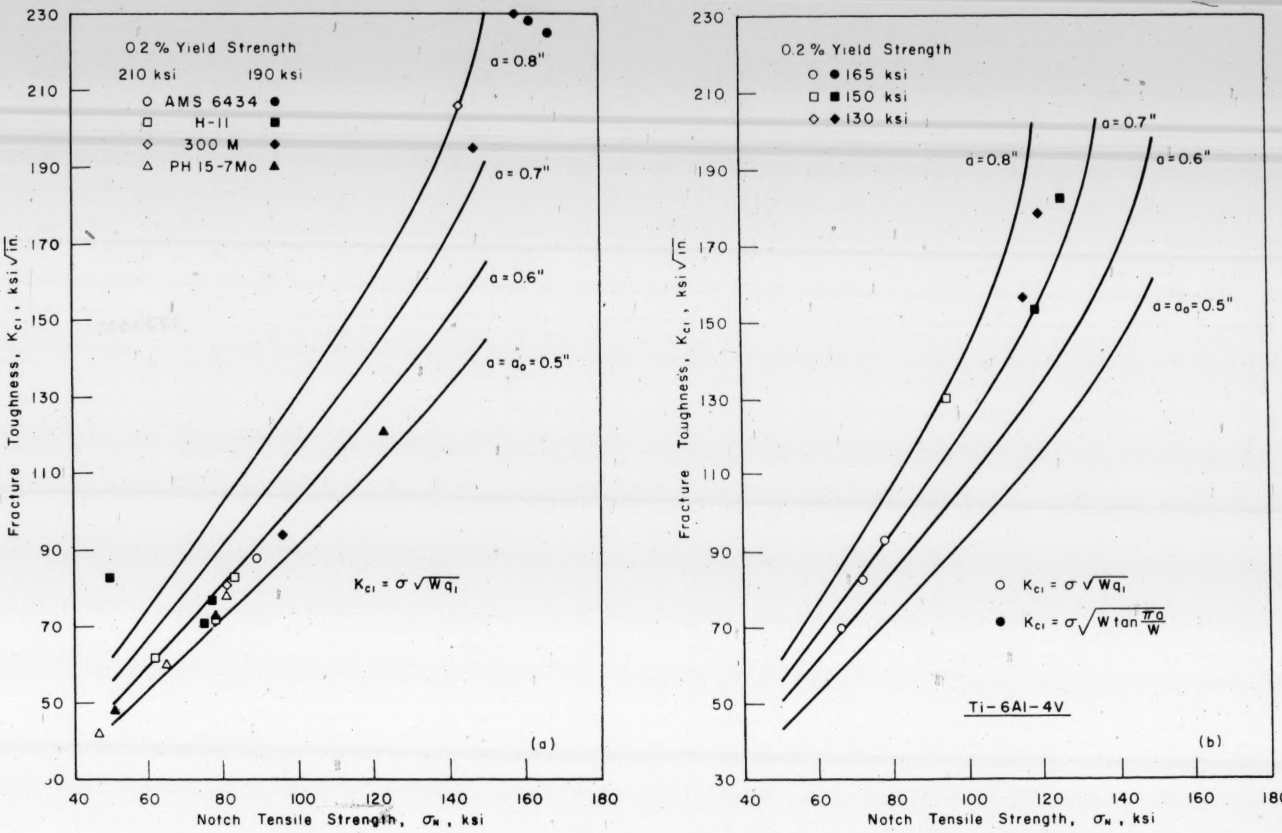
However, the gross fracture stress, σ, is directly related to the notch strength through the notch to unnotch area relationship, which is 2 to 3 since the notch length is one-third the width of the specimen. Then simply

$$\sigma = \tfrac{2}{3}\,\sigma_N \quad \text{.............} (2)$$

Substituting Eq 2 in Eq 1

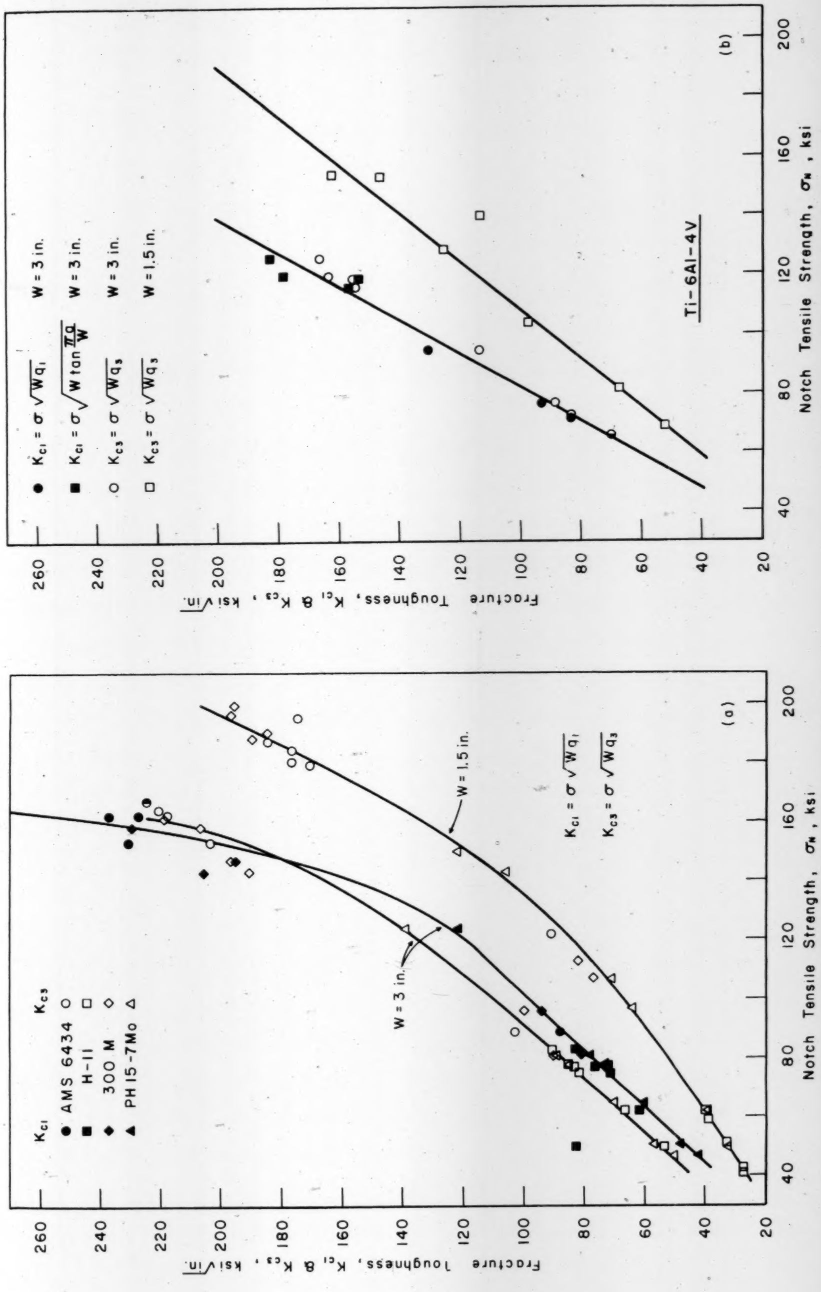
$$K_c = \tfrac{2}{3}\,\sigma_N\sqrt{wq} \quad \text{..........} (3)$$

Now using Eq 3, it is possible to con-



(*a*) Various steels. (*b*) 6Al-4V titanium alloy.

FIG. 4.—Notch Tensile Strength Versus Fracture Toughness Using 3-in. Wide Specimens.



(a) Various steels.

(b) 6Al-4V titanium alloy.

FIG. 5.—Comparison of Fracture Toughness Methods Versus Notch Tensile Strength.

struct curves which show the relationshp between K_c and σ_N. In order to accomplish this, it is necessary to assume values of slow crack extension, a, and the smooth bar tensile yield strength, σ_y. A value of 200 ksi was chosen for the yield strength and values for a were 0.5 ($a = a_0$, no slow crack extension), 0.6, 0.7, and 0.8. Figure 2 shown the result of this calculation. Thus it is clear that there is a near linear relation between fracture toughness, K_c, and notch strength, σ_N. Deviation from this linearity is encountered at higher notch strengths and larger slow crack extensions. Figure 2 is for the 3-in. wide specimen.

It is also possible to make similar calculations for the 1.5- and 1-in. wide specimens using Eq 3. This was done, assuming $a = a_0$ (no slow crack growth) and using the same σ_y as above, in order to show the relationship of σ_N and K_c for the various size specimens. The results of these calculations are shown in Fig. 3. Note the greater sensitivity of the 3-in. wide specimen forming almost an ideal 45-deg relationship. Clearly the 1.5- and 1-in. notch strengths have a great deal less sensitivity to fracture toughness and a much smaller range of useful values. This behavior is as would be expected for the stress gradient (**4,5**) increases, and the stress concentration factor, K_t, decreases as the specimen size decreases. The above is based on the assumption that K_c is a material constant. As can be seen from Fig. 3, the notch strength, σ_N, increases with decreasing specimen size. Thus, the narrow specimens are only suitable for testing brittle materials whereas the wide specimens can be useful over a wider range of K_c values since for accurate K_c determination the ratio of notch strength to yield strength must not exceed 0.8 (**6**).

The test results obtained in this investigation on the 3-in. specimen are compared to the σ_N versus K_c curves in Fig. 4 (*a*). It can be seen that the slow crack extensions range between $a = a_0$ and $a =$ 0.8. There is one unexplained point for H-11 steel.

A similar plot for the titanium alloy was constructed using $\sigma_y = 150$ ksi and is shown in Fig. 4 (*b*). It can be seen that the 165-ksi condition is associated with low notch strength plus a small amount of slow crack extension, which results in low fracture toughness values.

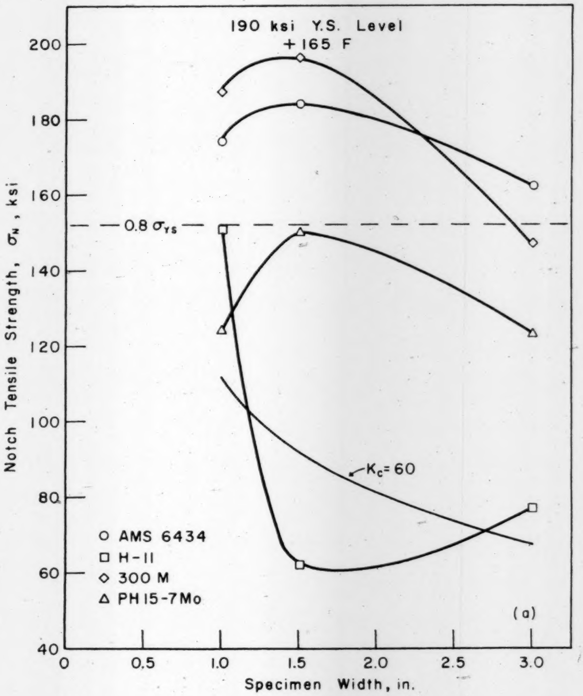
An interesting comparison between steels and the 6Al-4V titanium alloy is the greater slow crack extension and resultant fracture toughness for equivalent notch tensile strength which exists in the 150- and 130-ksi yield strength titanium. For example, in the steels a notch tensile strength of 120 ksi is associated with a slow crack extension and fracture toughness of approximately 0.6 and 120 ksi$\sqrt{\text{in.}}$ respectively, whereas for the titanium alloy the associated values are approximately 0.75 and 170 ksi$\sqrt{\text{in.}}$, respectively.

METHOD OF FRACTURE TOUGHNESS COMPUTATIONS

Since both ink stain and per cent shear measurements were made it is possible to compare fracture toughness values computed from these two measurements. In this investigation, the ink stain method, K_{c1}, and the per cent shear method, K_{c3}, were computed and the results are plotted in Figs. 5 (*a*) and (*b*). Also included in these figures are the K_{c3} (shear lip) values computed from the 1.5-in. wide specimens. This was done primarily to show the lower sensitivity of 1.5-in. specimens and the smaller range of use. This will be discussed further later in the report. As can be seen in Fig. 5 (*a*), the K_{c3} method results in a higher fracture toughness value than does the K_{c1} method up to a level of approximately 180 ksi$\sqrt{\text{in.}}$ Above this point it appears that K_{c1} values may be slightly higher.

Figure 5 (*b*) is a similar plot for 6Al-4V titanium,[3] and based upon limited data it appears that little difference exists between the two methods.

Numerous investigators, including references (1) and (7), have reported various trends between the K_{c1} and K_{c3} methods; however, as yet the exact reason seems to be lacking. Irwin and Srawley (8) have discussed the effect of the zone of plastic yielding near the leading edge of the crack and the associated plastic zone correction factor, $GE/2\pi \text{ YS}^2$. This is likely a contributing reason since this correction factor is numerically dependent upon material, strength level, and testing temperature. Others (7) feel that such differences can be attributed to the susceptibility of the staining method, K_{c1}, to human error during the process of employing the technique, making the necessary measurements, and interpreting the results.



(*a*) At +165 F.

FIG. 6.—Effect of Specimen Width on Notch Tensile Strength of Various Steels at the 190-ski Yield Strength Level. (Figure 6 continued on page 792.)

Concerning the K_{c3} method, the authors feel that the location where per cent shear is measured is of prime importance. Examination of numerous specimens revealed that the flat fracture width either varies or becomes zero (100 per cent shear) as the crack grows from the tip of the fatigue crack to the edge of the specimen. Therefore it can be seen that in either of these cases the selected value of per cent shear results

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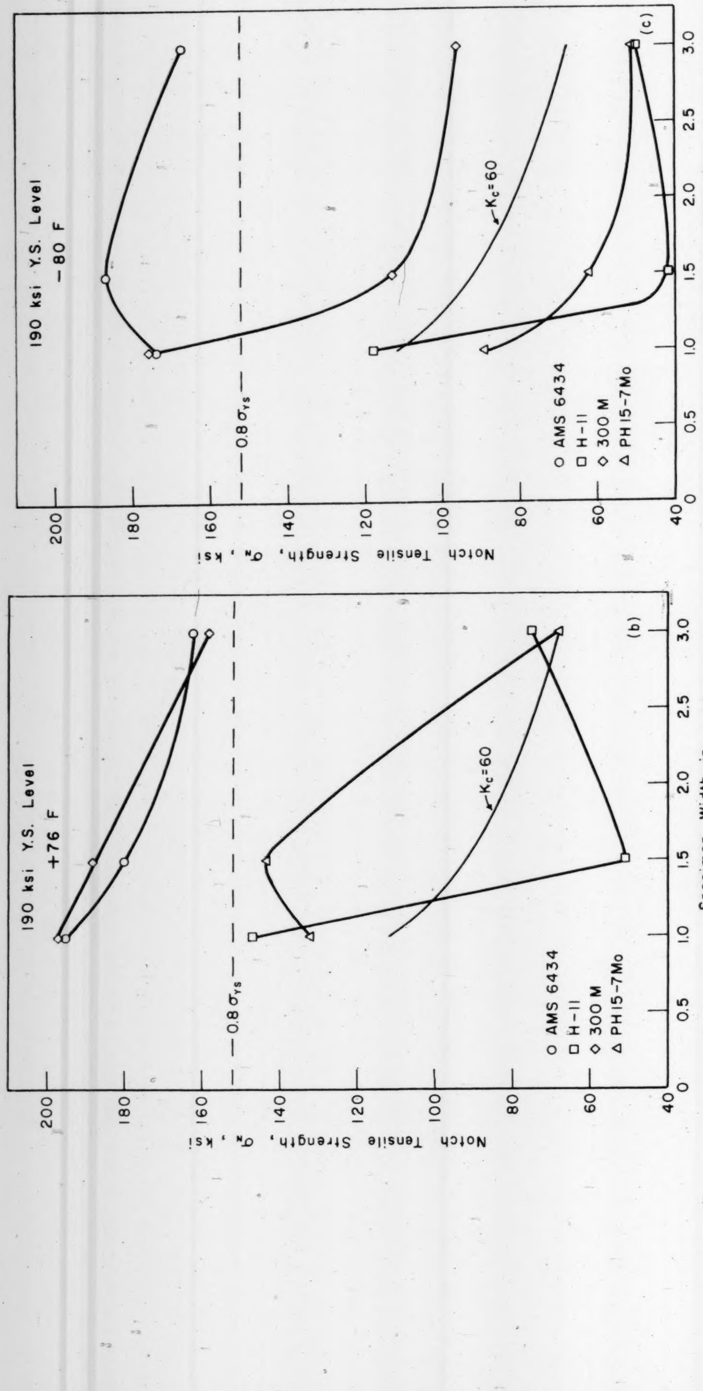
[3] In some cases q_1 fell outside the limits of the chart; therefore fracture toughness values were determined by the equation $K_{c1} = \sigma\sqrt{W \tan \pi a/W}$.

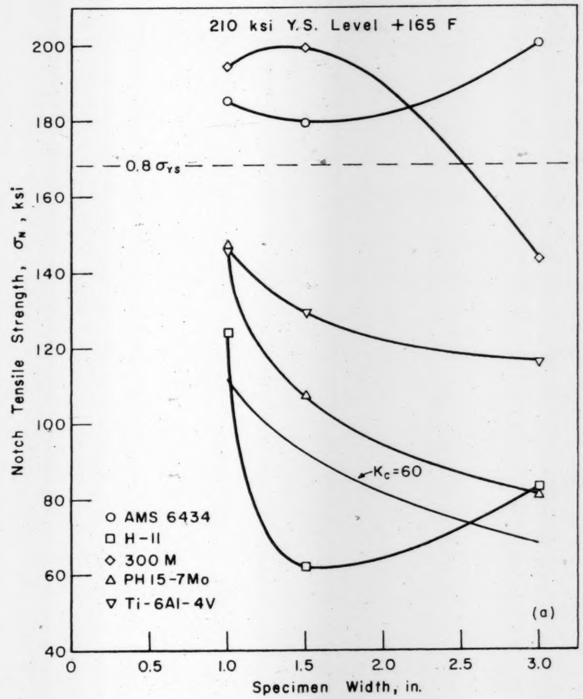


(*b*) At +76 F.

(*c*) At −80 F.

FIG. 6.—*Continued.*

in a K_{c3} which pertains only to a specific area along the fast crack growth portion of the fracture. Since fracture toughness is closely associated with slow crack extension, it is felt that K_{c3} values might be more realistic if per cent shear measurements were obtained at the termination point of slow crack extension.



(*a*) At +165 F.

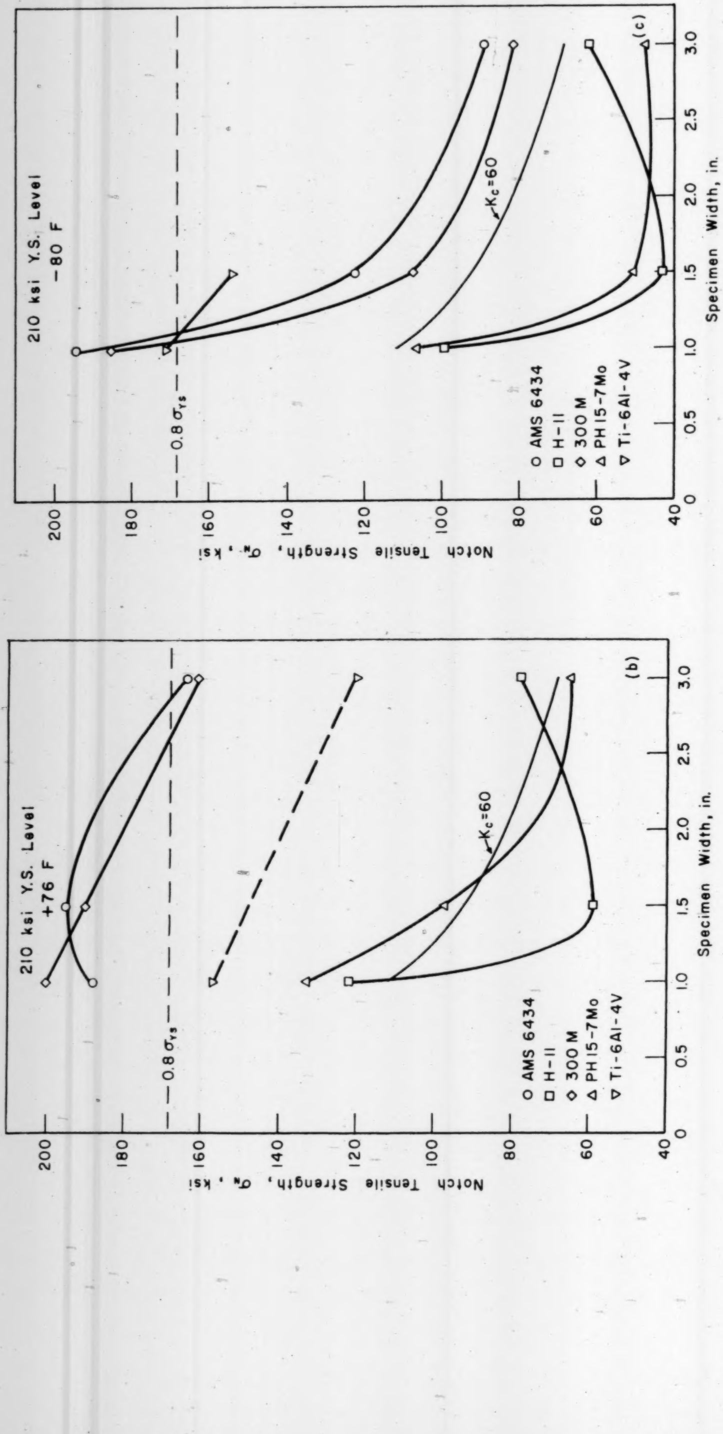
FIG. 7.—Effect of Specimen Width on Notch Tensile Strength of Various Materials at the 210-ksi Level. (Figure 7 continued on page 794.)

COMPARISON OF NOTCH STRENGTH AS A FUNCTION OF SPECIMEN WIDTH

Since K_c values were not obtained on the 1-in. wide specimens, the only way the various width specimens could be compared is on the basis of notch strength. As previously pointed out, this would be equivalent to comparison on the basis of K_c provided the notch strength did not exceed 0.8 of the yield strength.

Figures 6 (*a*) to (*c*) show plots of such data for the steels at the 190-ksi yield strength level and tested at +165, +76, and −80 F, respectively. Figures 7 (*a*) to (*c*) show plots of data obtained from the steels at the 210-ksi yield strength level at these same temperatures plus similar data from the 130-ksi yield strength level titanium alloy. (On a strength to density basis titanium of 130-ksi is approximately equivalent to 210-ksi steel.)

Steels at the 190-ksi yield strength level and tested at +165 F are shown in Fig. 6 (*a*). Data points for AMS 6434 and 300M steels fall either near



(b) At +76 F.

(c) At − 80 F.

FIG. 7.—*Continued.*

or above the 0.8 yield strength line in all cases whereas values for H-11 and PH 15-7 molybdenum alloys are below such line. The H-11 steel exhibits lowest σ_N in the 1.5-in. wide specimens; the reason for this is associated with the crack severity since this specimen was fatigue cracked prior to test. The H-11 is well known for its sensitivity to sharp cracks. The σ_N value for PH 15-7 molybdenum tends to be higher for the 1.5-in. specimen that for the 1-in. specimen. This may perhaps be accounted for by the difference in stress distribution in an edge and center notch specimen; however, the important aspect pointed out is that this material is not sensitive to a sharp notch at +165 F.

The AMS 6434 and 300M steels exhibit excellent toughness. It is actually difficult to compare materials on the basis of σ_N when such values approach the yield strength. Aspects such as gross yielding and a high degree of plastic deformation must be considered in these cases and such phenomena are more closely related to tensile properties rather than to toughness. Since this investigation is primarily concerned with toughness, little will be said about data which fall above the 0.8 yield strength level except to stipulate that such is representative of material which is not notch-sensitive.

Also plotted is the theoretical curve of $K_c = 60$ ksi$\sqrt{\text{in.}}$ (no slow crack extension) for the various specimen widths. This curve was obtained from the curves in Fig. 3.

Figures 6 (*b*) and (*c*) are similar plots of the same materials tested at +76 and −80 F respectively. As would be expected, the σ_N decreases as a function of decreased testing temperature. The H-11 material continues to show sensitivity to the severe notch created by fatigue cracking. It is clearly shown in Fig. 6 (*c*) that AMS 6434 possesses the greatest toughness of the materials investigated at the 190-ksi yield strength level.

Figures 7 (*a*) to (*c*) are plots covering the same temperature range for the steels at a 210-ksi yield strength level plus the 6Al-4V titanium alloy at a 130-ksi yield strength level. These plots illustrate that σ_N for the steels decreases with increased strength level plus decreased testing temperature. It is interesting to note that σ_N data for the titanium alloy tend to show an increase with decreased testing temperature. The H-11 steel is again sensitive to the severe notch created by fatigue cracking. It is interesting to point out that a transition evidently exists for AMS 6434 and 300M steels between +76 and −80 F. Again, it is clearly shown that the 3-in. wide specimen provides the best discrimination of notch sensitivity for all materials except H-11 steel.

SUMMARY AND CONCLUSIONS

The work reported in this investigation is a comparison of various tension specimens in the determination of notch sensitivity and fracture toughness of high-strength sheet materials. Specimens ranging from a 1-in. wide edge notch to a 3-in. wide center notch type were utilized in obtaining σ_N, K_{c1}, and K_{c3} data from AMS 6434, H-11, 300M, and PH 15-7 molybdenum steels plus 6Al-4V titanium alloy. Such data were obtained as a function of testing temperature and at two or more yield strength levels.

Based upon the observations from this investigation, the following conclusions can be drawn:

1. Brittle materials are characterized by low notch tensile strength and small amounts of slow crack extension which result in low values of fracture toughness, whereas ductile materials have high notch tensile strength and large amounts

of slow crack extension which lead to high values of fracture toughness. In this investigation the H-11 and PH 15-7 molybdenum steels can be considered brittle while AMS 6434 and 300M steels are of the ductile type. The 6Al-4V titanium alloy, based upon an equivalent strength to density ratio, tends to show intermediate values.

2. Specimen size is a very important criterion in the selection of material. Results in this study indicate that most materials exhibit increased notch sensitivity as specimen size increases from 1.0 to 3.0 in. An exception to this trend is evidenced by H-11 steel which shows greater sensitivity in the 1.5-in. wide fatigue-cracked specimen than in the 3-in. wide specimen. It was also shown that the obtainable fracture toughness range increases with specimen width, thus a narrow specimen can be used only for relatively brittle materials. Therefore it has been concluded that of the specimens considered in this investigation the 3-in. wide specimens generally offer the best test to measure notch sensitivity or fracture toughness.

3. In the steels investigated, fracture toughness values obtained by the per cent shear method, K_{c3}, are higher than those determined by the ink staining method, K_{c1}, to a level of approximately 180 ksi$\sqrt{\text{in.}}$ whereas above this point K_c values are higher. Little difference exists between the two methods for the titanium alloy. Possible explanations concern human errors in the ink stain technique and point of measurement in the per cent shear method.

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(1) "Fracture Testing of High-Strength Sheet Material, A Report of a Special ASTM Committee," *ASTM Bulletin*, No. 243, Jan., 1960, p. 29.

(2) "Fracture Testing of High-Strength Sheet Material, A Report of a Special ASTM Committee," *ASTM Bulletin*, No. 244, Feb., 1960, p. 18.

(3) "Current Tests for Evaluation of Fracture Toughness of Sheet Materials at High-Strength Levels," *DMIC Report 124*, Jan. 28, 1960.

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(5) B. M. Wundt, "A Unified Interpretation of Room-Temperature Strength of Notched Specimens as Influenced by Their Size," ASME Paper Number 59-MET-9, presented at Metals Engineering Conference, Albany, N. Y. April 29-May 3, 1959.

(6) G. R. Irwin, "Relation of Crack Toughness Measurements to Practical Applications," *Welding Journal Research Supplement*, Nov., 1962.

(7) H. W. Maynor, Jr., R. E. Mueller, and E. O. Jones, Jr., "Investigation of Mechanism of Failure of High-Strength Materials," Contract No. DA-1-01-009-ORD-889, Auburn Research Foundation of Auburn University, Jan. 12, 1962.

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DISCUSSION

Messrs. J. E. Srawley and W. F. Brown, Jr.[1] (*presented in written form*).—The authors have presented a considerable amount of notch data that are useful in comparing the results from three types of sheet specimens frequently used in fracture testing. In conducting the investigation an attempt was made to follow certain recommendations of the ASTM Fracture Committee as set forth in their first report. It is of interest to discuss some of the authors' observations in the light of more recent developments in fracture testing.

First it should be mentioned that the ASTM Fracture Committee does not specifically recommend "three general types of specimens, an edge-notch and two center-notch designs." For fracture toughness testing the first ASTM Committee report gives representative dimensions for both center-slotted and edge-notch specimens. These dimensions depend upon the thickness so that the width-to-thickness ratio lies between 16 and 45. All three specimens used by the authors are encompassed by the committee recommendations, provided the width-to-thickness requirements are met. The 1-in. wide edge notch specimen shown in Fig. 1(*a*) of the paper was recommended by the committee for screening, and for this purpose no width-to-thickness limitations were imposed.

The effect of specimen width on the notch strength, as shown in the authors' Figs. 6 and 7, is complicated by the fact that all specimen types did not have the same notch root radius. However, the data do indicate that a 1-in. wide specimen is not of sufficient size to discriminate between the lower yield strength alloy conditions. It may be noted that these are comparable in toughness with the recently developed maraging steels at higher strength levels. The ASTM Fracture Committee recognized that with recent improvements in the toughness level of structural sheet alloys, there is a need to increase the size of the screening specimen. For this reason a recently drafted proposed Tentative Method of Sharp Notch Testing of High Strength Sheet Materials recommends a 3-in. wide edge or center notch screening specimen.

A forthcoming report of the ASTM Fracture Committee will suggest that fracture toughness specimens contain fatigue cracks and that staining fluids be abandoned for the purpose of slow crack extension measurement. These modifications reflect considerable additional experience in fracture testing since publication of the First Committee Report. In this regard it should be noted that the Third Committee Report[2] presented data showing that the apparent K_c values for H-11 (1000 F temper) were lower for fatigue cracks than for 0.001-in. radius machined notches. The authors' data confirm this observation and indicate similar effects would be observed in other relatively brittle alloys. Thus, according to their Table IV, it appears that when K_{c3} values could properly be obtained with 1.5-in. wide center-fatigue crack specimens,[3] these values were lower than those given by the 3-in. wide center-slotted specimens which were not fatigue cracked. Regarding substitution of other methods for ink staining in determining slow crack extension, attention should be given to the paper by Anctil et al[4] which describes an electrical method suitable for this purpose.

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[1] Head, Fracture Section, and Chief, Strength of Materials Branch, respectively, National Aeronautics and Space Administration, Lewis Research Center, Cleveland, Ohio.

[2] "Fracture Testing of High-Strength Sheet Materials: Third Report of a Special ASTM Committee," *Materials Research & Standards*, Vol. 1, No. 11, Nov., 1961, p. 877.

The writers personally believe that the use of per cent shear in the calculation of fracture toughness should be discouraged because the resulting K_{c3} values may not represent the true toughness of the material. Difficulties arise when appreciable slow crack extension occurs with essentially flat fractures, such as is observed for some titanium alloys; or when low toughness conditions exhibit oblique shear fractures, a behavior observed in some silicon-modified low-alloy steels.

MESSRS. C. F. HICKEY, JR. AND F. R. LARSON.—The authors wish to thank Messrs. J. E. Srawley and W. F. Brown, Jr. for their written discussion of this paper. We concur with their comments.

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[3] It is assumed that yielding on the net section did not occur if the ratio of net section stress-to-yield strength was less than 0.5.

[4] A. A. Anctil, E. B. Kula, and E. DiCesare, "Electric Potential Technique for Determining Slow Crack Growth," see p. 799.

ELECTRIC POTENTIAL TECHNIQUE FOR DETERMINING SLOW CRACK GROWTH*

By A. A. Anctil,[1] E. B. Kula,[1] and E. DiCesare[1]

Summary

Slow crack growth was followed as a function of applied load during fracture toughness testing of high-strength sheet materials by means of continuous measurement of electric potential field changes across the crack. The instrument employed is a commercially available milliohmmeter and uses the ammeter-voltmeter measurement method with a minimum of current passing through the test specimen. Voltage drop across the crack, rather than resistance, is measured. Electronic and mechanical testing techniques are described.

Typical potential field distribution diagrams illustrate the potential change with crack growth. A single calibration curve for crack size versus potential drop may be obtained independent of material and specimen configuration by maintaining geometrical similarity of current and potential contacts. Calibration data are readily obtainable for any specimen design by duplicating the specimen in aluminum foil and simulating crack growth with a razor.

Slow crack growth has been followed in 4340 and 300M steels heat treated to several strength levels as well as cold rolled and aged after heat treatment. Typical curves of load versus potential drop relate the various stages in the fracturing process.

In evaluating high-strength materials, it has become apparent that the standard unnotched tension test does not adequately predict the behavior of the material in actual service. The presence of sharp notches or cracks has been found to lower the performance of these materials severely. Recognizing this, the special ASTM Committee on Fracture Testing of High-Strength Metallic Materials has recommended screening tests using very sharp notches (**1**).[2] A complete analysis of the fracture of such specimens by fracture mechanics requires a knowledge not only of the load and stress intensity at which rapid crack propagation starts, but also of the size which the crack achieved by slow crack growth at the point of instability.

Several techniques have been suggested for determining one or both of the above in sheet specimens. These include visual observation and ink staining (**1**), photographic (**2**), sonic (**3**), strain gage (**4**), compliance gage (**5**), and electrical resistance (**6**) methods. Initially, ink staining achieved greatest usage, but this technique suffers the disadvantage of being somewhat difficult to use, especially at other than room temperature. The ink tends to obscure the appearance of the fracture; but most important, the presence of the ink may itself alter the resistance to crack growth (**7**). The compliance gage is currently finding greater favor (**5**,**8**). Although capable of yielding continuous load versus crack growth data, it is relatively insensitive at small crack sizes; the modulus of elasticity must be known at the temperature of test; and the gage itself can be damaged during fracture. Steigerwald and Hanna used a Kelvin double bridge for measuring resistance (**6**). This is capable of measuring very low resistances with high precision. Dis-

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* The statements and opinions expressed in this article are those of the authors and do not necessarily indicate the views or policy of the U. S. Army Materiel Command.

[1] Mechanical Metallurgy Branch, U. S. Army Materials Research Agency, Watertown Arsenal, Watertown, Mass.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

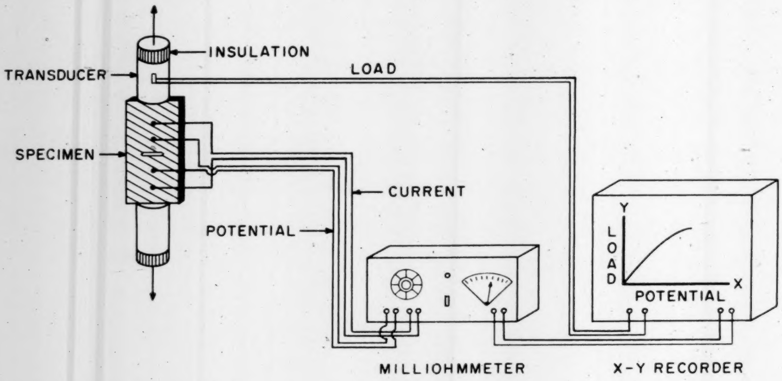

FIG. 1.—Schematic Drawing of Electric Potential Measurement and Recording Setup.

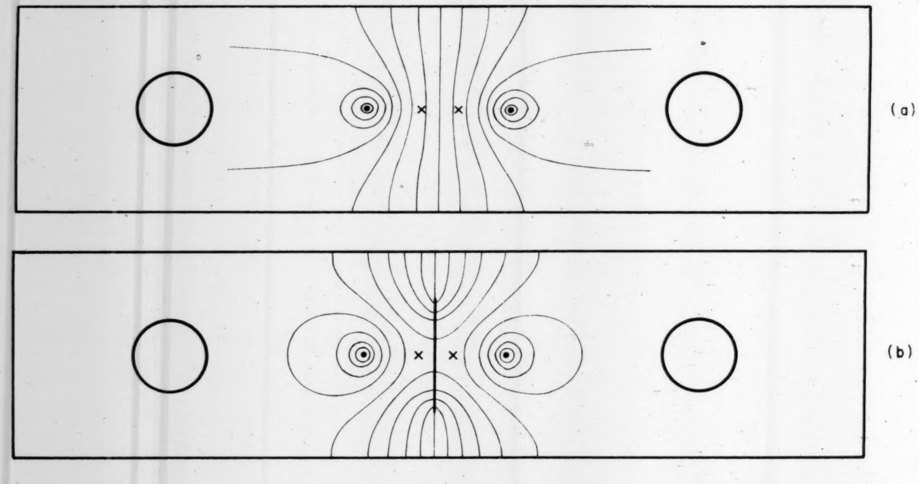

FIG. 2.—Electric Potential Field Distributions (Equipotential Lines) (*a*) Without a Crack and (*b*) with a Crack.

advantages are that the resistance has to be recorded manually, and heating of the specimen can occur because of the very high current used. Also, a separate calibration must be made for each material.

and, as in the case of the compliance gage, the complete course of the slow crack growth can be followed.

In the present investigation, a commercially available milliohmmeter was used (12) which measures resistance by the ammeter-voltmeter method. The instrument uses an a-c measuring system which minimized current through the specimen and eliminates errors due to thermal electromotive forces. The current is only 100 ma at full scale on the

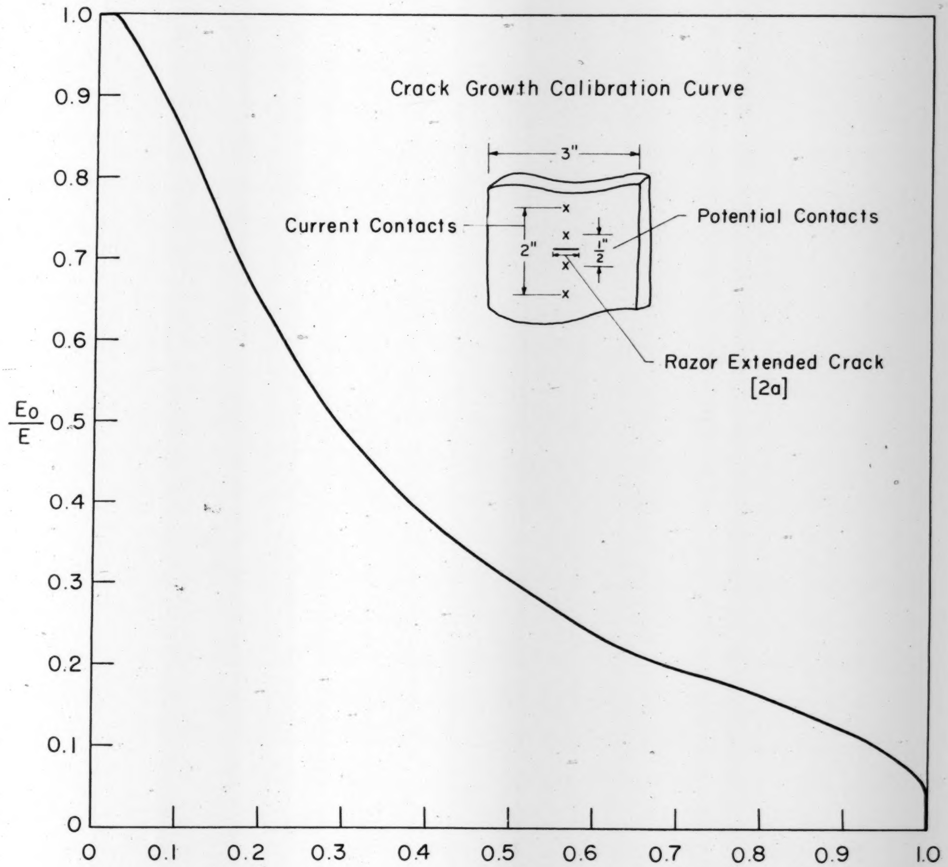

FIG. 3.—Calibration Curve Relating Electric Potential and Crack Size for Center Notched Specimens.

CRACK SIZE DETERMINATION

An electrical technique would seem to offer several advantages for studying crack sizes (6,9–11). An electrical signal can be fed easily into an *X*-*Y* recorder and be recorded together with the load

0.001-ohm range. Response time is 0.25 sec to 90 per cent full scale, which is adequate for following slow crack growth. Contact resistance is minimized by using four terminal connections. The experimental setup is shown in Fig. 1.

Laplace's equation, $\nabla^2\phi = 0$, is the general equation describing the electric potential field in a specimen for any boundary conditions—specimen geometry, crack size, and location of current leads. Ohm's law then relates the potential to the resistivity and current density. Several methods for solving Laplace's equation can be found in standard texts (**13**), but the direct experimental method seems best for a complicated specimen geometry. In this case a model of the test specimen was made of 0.002-in. aluminum foil. The foil was found to have a resistance high enough to be in the range of the milliohmmeter. The foil can conveniently be cut to size and the crack extended by a razor blade. Other materials including actual test specimens may be used. Experimentally determined potential fields are shown in Fig. 2 for specimen configurations with and without a crack. The solid circles show the position of the current leads, and the thin black lines are the equipotential lines determined by the voltage leads.

Note that the potential changes with crack size are greatest in the region along the center line of the specimen and near the crack. The potential is also somewhat less sensitive to exact position of the leads in this region. For following the course of crack growth, the potential was measured at points marked X. It should be mentioned that the actual resistance of the specimen is not measured. The milliohmmeter supplies a constant current and the two potential leads measure a voltage drop which is converted to resistance. If all the current passed between the potential leads, the specimen resistance would be measured. However, since all the current does not flow between the potential leads, it is best to speak of potential differences rather than of resistances.

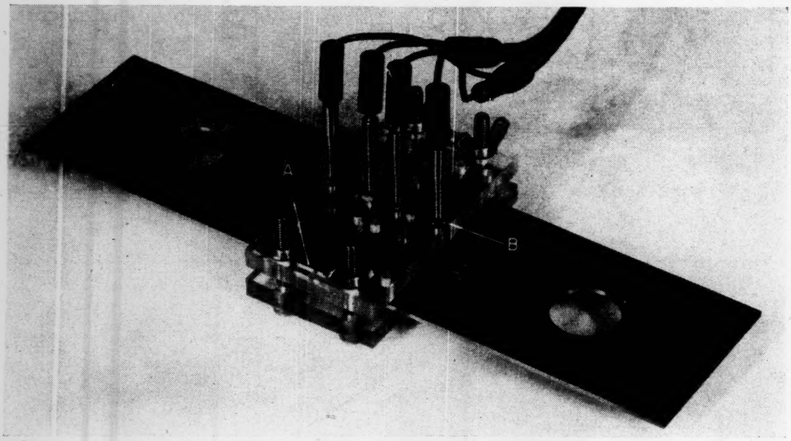

FIG. 4.—Gage for Measurement of Electric Potential.

With a given geometry and constant current, the potential drop across the crack will be an accurate indication of the size of the crack. The magnitude of the potential drop will also depend on the resistivity of the material. In order to eliminate the effects of such variables as resistivity, specimen thickness, and test temperature, a ratio of the potential drop without a crack to the potential drop with a crack, E_0/E, is taken. Figure 3 shows this ratio of voltages as a function of the crack size ratio, $2a/W$, for the indicated geometry. For crack sizes of interest, the measurements lie on the central portion of the curve.

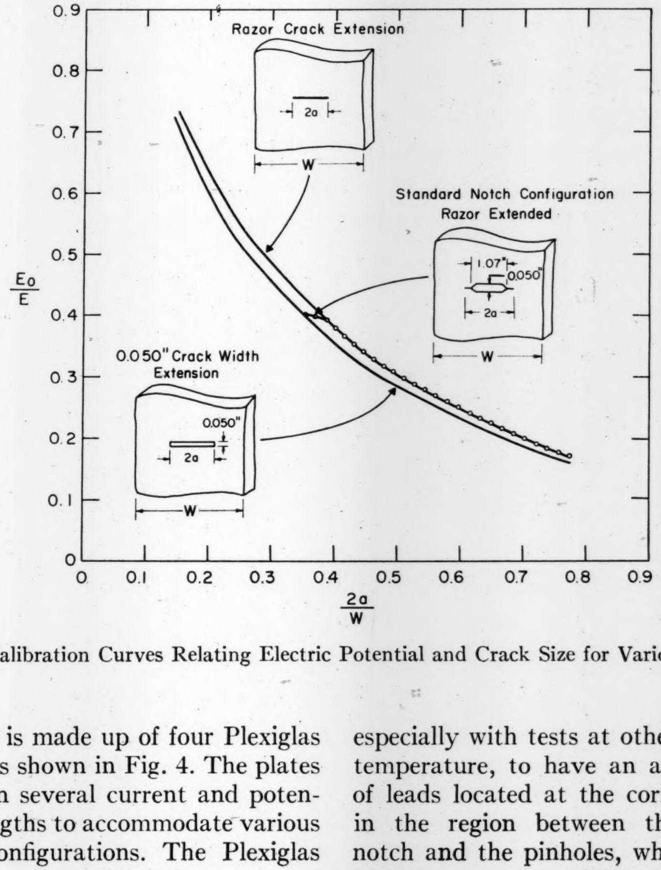

Fig. 5.—Calibration Curves Relating Electric Potential and Crack Size for Various Crack Configurations.

The gage is made up of four Plexiglas rectangles as shown in Fig. 4. The plates may contain several current and potential gage lengths to accommodate various specimen configurations. The Plexiglas could be cut out to allow observation of fatigue crack opening and subsequent slow growth during testing. Note the small vertical alignment pin, *A*, and lock nuts, *B*. Electrical contacts with the specimen are made through pointed screw heads. For elevated temperature use, some other material insulated from the specimen leads could be used for the plates.

The distance between the current leads is such that the uncracked potential drop, E_0, can conveniently be measured in the region between the machined notch and one of the pinholes before or after the test. Instead of physically moving the gage it may be more convenient, especially with tests at other than room temperature, to have an additional set of leads located at the correct distance in the region between the machined notch and the pinholes, which could be connected to the milliohmmeter immediately before and after the test.

The calibration curve of Fig. 3 was determined with an aluminum foil model of a 3-in. specimen. The same curve is valid for other specimen widths provided the distances between the crack and the current and potential leads are changed in direct proportion to the width change. This curve can also be used for edge notched specimens since the potential fields are the same as would be obtained by taking the diagram of Fig. 2(*b*), cutting it longitudinally along the center line, and shifting the two halves so that the edges of the center notch specimen become the center line of the edge notched specimen. In this case the potential leads can be placed at either edge, but the current leads must be divided and placed at both edges (four points).

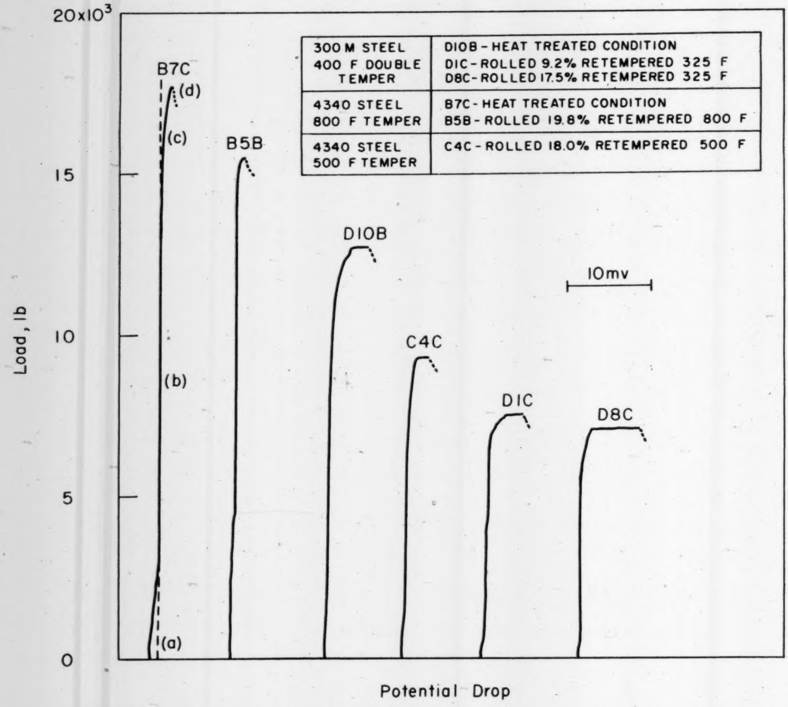

FIG. 6.—Typical Load-Potential Drop Curves for 4340 and 300M Steels.

On an actual specimen the machined notch is not razor sharp but has a finite width, since material has been removed. Because of this, the voltage drop is greater at any given crack size and the ratio E_0/E would fall somewhat below the curve shown in Fig. 3. Three calibration curves are shown in Fig. 5 to illustrate this point. The upper curve was obtained using a razor sharp crack. In the lower curve the notch was extended, maintaining a constant width of 0.050 in. The transitional portion refers to the start of an actual calibration curve with an initial notch 0.050 in. wide extended by a razor cut, simulating a machined notch extended first by fatiguing and then propagating during testing. The initial ratio E_0/E falls on or near the lower curve, then approaches the razor calibration curve rapidly and coincides with it. An extension of the 1.07-in. machined notch by only 0.110 in. is sufficient to remove the effect of the finite width of the initial machined notch.

Since the shape of the initial machined notch is not standardized, the best procedure is to determine a calibration curve starting with the particular machined notch used. The crack is then extended by a razor, and the potential drop across the crack is measured. This procedure is advisable also since a location of current and potential leads other than that shown in Fig. 2 may be necessary.

TABLE 1.—TENSILE AND FRACTURE TOUGHNESS DATA.

Specimen and Material	Processing		Tensile Data				Fracture Data				
	Retempered, deg F	Per Cent Reduction	Ultimate Tensile Strength, ksi	0.2 Per Cent Yield Strength, ksi	$2a_0$, in.	$2a$, in.	Per Cent Shear	F_{net}	F_{net}/0.2 Per Cent Yield Strength Ratio	K_{c_v}, ksi $\sqrt{in.}$	K_{c_3}, ksi $\sqrt{in.}$
4340 steel tempered at 800 F:											
B7C	...	0	204	193	0.697	0.836	100	183	0.95	189	216
B1C	800	19.3	209	207	0.733	0.910	93	178	0.86	180	199
B5B	800	19.8	215	214	0.698	0.830	100	168	0.78	154	186
B3B	800	30.9	218	217	0.699	0.756	90	176	0.81	150	193
B3C	800	30.1	218	217	0.667	0.946	100	161	0.74	170	178
4340 steel tempered at 500 F:											
C7C	...	0	255	221	0.693	0.934	90	179	0.81	198	204
C4C	500	18.6	279	279	0.704	0.920	100	100	0.36	88.4	104
300M steel tempered at 400 F:											
D10B	...	0	284	229	0.682	0.868	100	142	0.62	129	152
D10C	...	0	284	229	0.684	0.900	100	145	0.63	136	151
D2C	325	6.4	310	297	0.766	0.936	88	111	0.38	94.7	113
D4B	325	7.9	320	310	0.637	0.880	76	89.3	0.29	79.4	88.7
D4C	325	7.9	320	310	0.669	0.874	92	95.0	0.31	82.3	97.0
D1C	325	9.2	316	300	0.703	0.944	75	84.5	0.28	75.1	79.8
D3B	325	9.6	321	314	0.674	0.806	79	69.0	0.22	55.6	74.8
D3C	325	8.3	321	314	0.677	0.994	79	69.4	0.22	65.2	69.0
D8C	325	17.5	338	338	0.562	1.040	71	72.8	0.22	77.3	70.7

Note.—Specimen thickness 0.070 in., width 2.0 in., fatigue cracked.
All specimens rolled at room temperature.
Heat treatments given: 300M—1625 F for 1 hr, oil quenched; tempered 400 + 400 F for 1 hr.
4340—1700 F for 1 hr, air cooled; 1550 F for 1 hr, oil quenched.

The accuracy of the electric potential method was substantiated in two ways, both of which gave good agreement. The measured ratio, E_0/E, at the initiation of the elastic portion of the load-potential drop curve gave a value of crack size $2a_0/W$ using the calibration curve (Fig. 5). This value agreed very closely with the measured fatigue crack length. A further check was made by comparing the crack length measured on heat-tinted specimens with that determined by the electric potential method for the same specimens. Agreement was within 0.015 in.; however, an accurate measurement of the heat-tinted crack length was difficult because of the lack of a sharp crack front delineation and because of its shape.

CALCULATION OF FRACTURE TOUGHNESS

In an actual test, the load is recorded as a function of the voltage drop across the crack. Typical curves, similar in shape to those shown by Steigerwald and Hanna (6), are shown in Fig. 6. First there is a reversible voltage change (Fig. 6, specimen *B7C*) during elastic loading. This consists of an initial nonlinear portion, *a*, due to opening of the fatigue crack, followed by a linear portion, *b*, caused by the effect of stress on the resistivity. The curve deviates from linearity at *c* when plastic deformation or crack extension occurs and terminates at the start of fast fracture, *d*. In these high-strength steels changes due to plastic deformation can be neglected, and the measured deviation from the straight line portion can be attributed to crack extension. By expanding the potential drop range (millivolts) it is possible to determine crack initiation stress and type of discontinuous crack growth. Crack growth rates can be determined plotting potential drop and load versus time.

The total potential change due to slow crack growth may be determined in a similar manner as that used to determine yield strength at a given offset percentage from an engineering stress-strain curve. The irreversible change is added to the voltage drop across the machined notch before testing plus an increment for the fatigue crack opening during testing, to yield E_f, the total voltage drop across the crack in the absence of stress at the onset of rapid crack propagation. The ratio E_0/E_f is plotted on the calibration curve (Fig. 5), and the crack length, $2a$, is determined from the abscissa.

This technique for measuring crack size has been used for determining the fracture toughness of 300M and 4340 steels that have been strengthened thermomechanically. Three different base strength levels are represented—300M tempered at 400 F, and 4340 tempered at 500 F and 800 F. In the tempered condition the steels were rolled various percentages at room temperature and then retempered at or slightly below the original tempering temperature. Considerable strength improvements arise from such a treatment. Heat treatments and rolling reductions are shown in Table 1, together with smooth tensile and fracture toughness data. Initial thicknesses were varied and final thicknesses held constant so that all testing was carried out on sheet 0.070 in. thick. Fracture toughness specimens were 2 in. wide and fatigue cracked.

The critical stress intensity factor, K_c, was calculated both by using the experimentally determined value of $2a$ to yield K_{c_v}, and by the per cent shear method to obtain K_{c_3}. The net fracture stress was always less than the yield strength. The data in Table 1 show that K_{c_v} is a more conservative estimate of fracture toughness in almost all cases.

In measuring the flat fracture for the K_{c_3} calculations, little or no flat fracture was found for most of the specimens when measured between the recommended 1 and $2B$ from the edge of the specimen; however, the amount of flat fracture was considerably greater closer to the machined notch where rapid crack propagation started. The higher values of K_{c_3} may therefore be associated with this variation in fracture appearance across the specimen width.

The results were interesting in that many of the load-voltage drop curves contained one or more portions where the crack extended under constant load as in series *D* and *C* in Fig. 6. Examination during the test revealed that this crack extension occurred very rapidly compared to the extension taking place under increasing load. Several examples were noted where this accelerated growth preceded failure. Potential changes as a function of time substantiate this measurable rate increase. This type of discontinuous crack growth was noted most frequently in the 300M steel tempered at 400 F, occasionally in 500 F tempered 4340, and not at all in the latter steel tempered at 800 F. This feature of variable crack growth appears to be associated with lower toughness levels. In some cases it was possible to relate these rapid crack extensions to steps in the fracture surface, although in many cases no correlation with the fracture surface was evident.

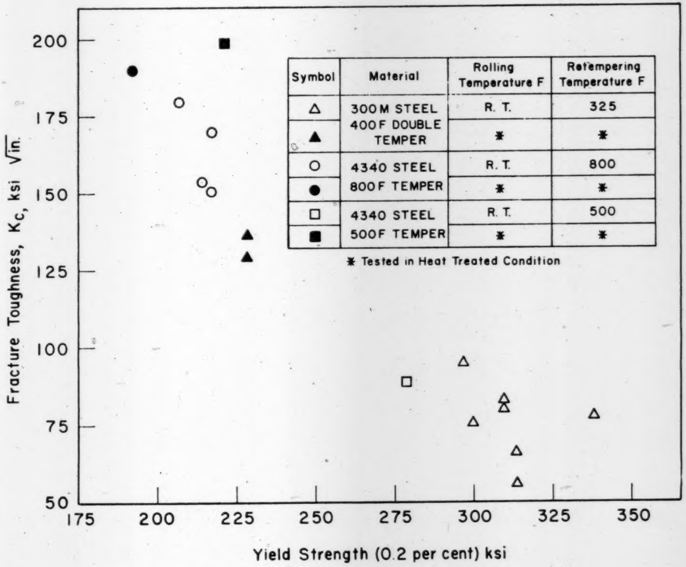

FIG. 7.—Variation of Fracture Toughness with Yield Strength for 4340 and 300M Steels.

The effect of rolling and retempering the quenched-and-tempered steels was to increase the yield and tensile strengths markedly. The increase was greater the higher the initial strength level of the steel. With sufficiently high reduction, the yield strength equaled the tensile strength. As might be expected, the values of fracture toughness decreased as the strength level increased. Figure 7 shows the values of K_{c_v} from Table 1 plotted against the yield strengths. There is a general scatter band showing the decrease in toughness with increase in strength level. Although it had been hoped that certain thermomechanical treatments would show better strength-toughness combinations than others, the scatter in the data precludes such conclusions.

SUMMARY

Slow crack growth in sheet tension specimens can be followed by measurement of the electric potential across the crack. The signal from a commercially available milliohmmeter is fed into a recorder to obtain load-potential drop or potential drop-time curves. Calibration of the potential drop with crack size can be made with measurements on aluminum foil cut to the shape of the specimen and with the crack extended by cutting with a razor. The calibration procedure is applicable to any type of potential measurement circuit. This method of determining crack size offers advantages over other methods in that it is relatively easy to make the measurements, the slow crack growth can be continuously followed, and a calibration of the potential drop with crack size can readily be made.

Using this technique, fracture toughness was determined in 300M and 4340 steels which, in the quenched-and-tempered condition, had been cold rolled and aged. Although significant strength increases were obtained by this treatment, the fracture toughness was severely lowered.

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EFFECTS OF SEVERAL PRODUCTION AND FABRICATION VARIABLES ON SHARP NOTCH PROPERTIES OF 5Al-2.5Sn TITANIUM ALLOY SHEET AT LIQUID HYDROGEN TEMPERATURE

By J. L. Shannon, Jr.,[1] and W. F. Brown, Jr.[1]

At liquid hydrogen temperature the strongest weldable aluminum alloys have yield strength to density ratios less than 900,000 in. (**1,2**).[2] If higher strength to density ratios are desired either titanium alloys or cold-rolled metastable stainless steels must be employed.

Metastable stainless steels cold rolled sufficiently to give −423 F yield strength to density values above 1,000,000 in. possess a number of characteristics which make them marginal for use in liquid hydrogen tankage. Thus, as previously shown (**3,4**), AISI Type 301 stainless steel exhibits a substantial decrease in crack tolerance as the temperature is reduced from −320 to −423 F. Both AISI Type 301 and Type 304 steels show high directionality of fracture toughness due to heavy cold rolling with the transverse toughness being relatively poor over the temperature range from +75 to −423 F (**5**). Furthermore, welds in metastable stainless steels are transformed to essentially 100 per cent martensite by very small plastic strains at −423 F and such transformed regions can act as crack starters.

Titanium alloys, like the stainless steels, exhibit a decrease in crack tolerance as the test temperature is reduced from −320 to −423 F. However, if the impurity content is sufficiently low the annealed alpha alloy, 5Al-2.5Sn titanium, has a better resistance to crack propagation at −423 F than AISI Type 301 at the same yield strength level (**3**). Additional advantages result from the use of this alloy in the annealed condition: there is essentially no directionality of the sharp notch properties, and welds have approximately the same strength as the parent metal and are stable at low temperatures. However, very little is known regarding the influence of various alloy production and fabrication variables on the fracture toughness of 5Al-2.5Sn titanium alloy sheet and such knowledge is necessary for rational hardware design.

The object of the present investigation was to determine the influence of the following alloy production and fabrication variables on the smooth and sharp notch strength of 5Al-2.5Sn titanium alloy sheet: (1) sheet thickness; (2) small amounts of cold rolling such as might be applied in flattening; (3) stretching in uniaxial tension; (4) cooling rate from the annealing temperature; and (5) fusion welding. Investigations of sheet thickness involved tests over a range of low temperatures on material at three interstitial levels. The majority of the remaining variables were studied at −423 F using a low-interstitial composition typical of that which might be specified for liquid hydrogen service.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

TEST MATERIAL

Four heats of 5Al-2.5Sn titanium alloy were investigated having the compositions shown in Table 1. The heats designated as normal interstitial had impurity contents typical of commercial grade alloy. The remaining two heats were produced by using a low-hardness sponge with no scrap additions. These are designated as low interstitial I and low interstitial II. The first was an experimental heat and the second a commercial heat produced to General Dynamics/Astronautics specification No. ES 0-71010. This specification was designed to limit the interstitial and iron content to the lowest values consistent with reasonable production costs. Both of the low-interstitial heats had closely matching impurity contents with the exception of nitrogen and hydrogen, which were significantly higher in the commercial heat (low interstitial II).

TABLE 1.—COMPOSITION AND HEAT TREATMENT OF 5Al-2.5Sn TITANIUM SHEET ALLOYS.

Interstitial Content Designation	Republic Steel Corp. Heat	Composition, weight per cent[a]							Thickness, in.	Annealing Treatment
		Aluminum	Tin	Carbon	Nitrogen	Oxygen	Hydrogen, ppm	Iron		
Low I.....	XT 70025	4.88	2.45	0.029	0.004	0.103	8.0	0.120	0.015, 0.025, 0.063, 0.080	1500 F, 45 min (vacuum), furnace cool[b]
Low II....	3960328	5.28	2.56	0.028	0.010	0.100	40.0[d]	0.110	0.010, 0.025, 0.060, 0.250	1500 F, 2 hr (argon), furnace cool[c]
Normal...	3930031	5.28	2.59	0.038	0.009	0.180	8.0	0.160	0.015, 0.025, 0.063	1500 F, 45 min (vacuum), furnace cool[b]
Normal...	3950620	5.20	2.48	0.030	0.014	0.200	70.0	0.170	0.125	1500 F, 2 hr (argon), furnace cool[c]

[a] Furnished by supplier unless otherwise noted.
[b] Annealed by alloy supplier.
[c] Reannealed by NASA.
[d] Determined by NASA.

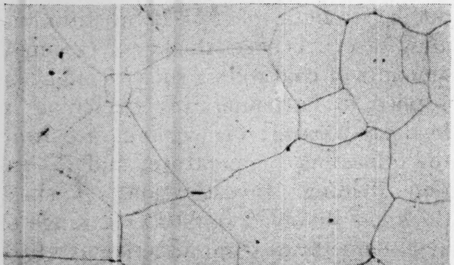

FIG. 1.—Typical Microstructure of 5Al-2.5Sn Titanium Alloy Annealed at 1500 F (×500); Etch 30 Lactic Acid, 10 HNO_3, 5 HF.

As indicated in Table 1, all sheet was given a 1500 F anneal in either vacuum or argon and furnace cooled. Material annealed in the authors' laboratory had previously received lower temperature annealing at the mill which in some cases did not entirely remove all evidences of cold work. It is to be noted that after the annealing treatments of Table 1, the microstructure showed no evidence of cold work. A typical example is given in Fig. 1 which shows equiaxed grains characteristic of annealing in the alpha field.

TEST PROCEDURE

The material conditions and variables investigated are outlined in Table 2. Details of the rolling, stretching, and welding procedures are given in the Appendix.[3] Sharp-edge notch tests were used to establish the influence of the various material conditions on the fracture characteristics at cryogenic temperature. The following section presents details concerning specimen preparation and testing procedure as well as the method of presenting the data.

TABLE 2.—MATERIAL CONDITIONS AND VARIABLES INVESTIGATED.

Variable Investigated	Material Condition	Interstitial Level	Sheet Thickness, in.	Test Temperature, deg F
Test temperature	1500 F, 2 hr (argon), air cool	Low II	0.010, 0.025, 0.060, 0.250	Room temperature, −110, −320, −423
	Annealed (see Table 1)	Normal	0.015, 0.025, 0.063, 0.125	Room temperature, −110, −320, −423
Sheet thickness	Annealed (see Table 1)	Low I	0.015, 0.025, 0.063, 0.080	Room temperature, −110, −320, −423
	Annealed (see Table 1)	Low II	0.010, 0.025, 0.060, 0.250	−423
	Annealed (see Table 1)	Normal	0.015, 0.025, 0.063, 0.125	Room temperature, −110, −320, −423
Room temperature stretching	Stretch 0 to 16 per cent	Low I	0.025,[a] 0.063[a]	−423
	Stretch 0 to 20 per cent	Low II	0.010,[a] 0.025,[a] 0.060[b]	−423
Cold rolling	Roll 0 to 10 per cent	Low II	0.010,[a] 0.025[a]	−423
Welding	Tungsten inert gas, fusion weld	Low I	0.025[a]	Room temperature −423
		Normal	0.025,[a] 0.125[a]	Room temperature −423
Annealing treatment	1500 F, 2 hr (argon), air cool	Low II	0.010, 0.025, 0.060, 0.250	−423
	Annealed (see Table 1)	Low II	0.010, 0.025, 0.060, 0.250	−423

[a] Initially in the annealed condition shown in Table 1.

[b] Initially mill annealed; 1400 F, air cool.

Specimen Preparation and Testing:

The smooth and sharp-edge notch tension specimens are shown in Fig. 2. Details regarding the design and preparation of sharp-edge notch tension specimens for screening purposes are described in a report of the ASTM Committee on Fracture Testing of High-Strength Sheet Materials (6). The notch specimen employed in this investigation is basically that described by the ASTM committee, except that notch radii were held to 0.0007 in. maximum instead of the 0.001 in. mentioned in the committee report. With the exception of the welded speci-

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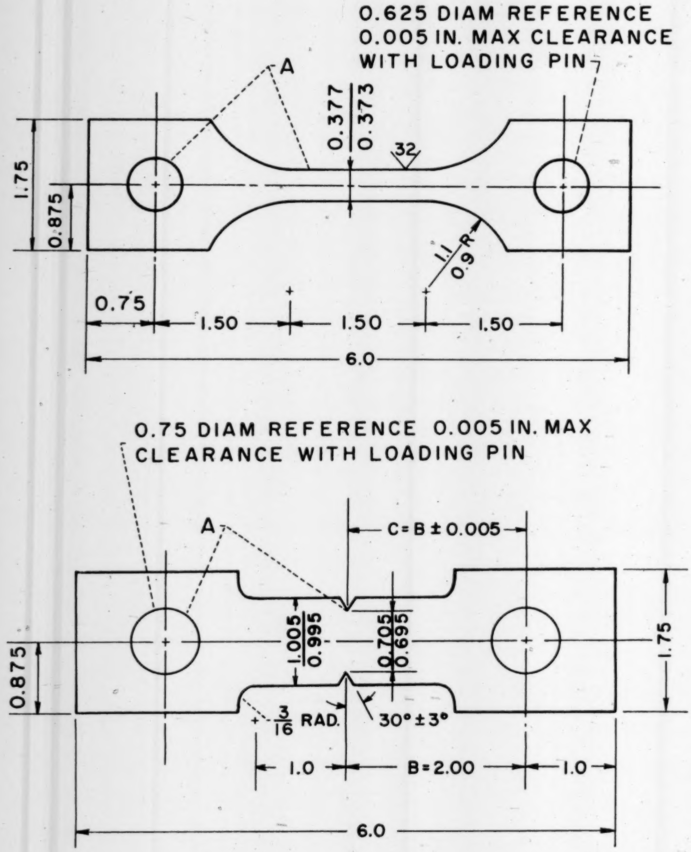
[3] See p. 824.


0.625 DIAM REFERENCE
0.005 IN. MAX CLEARANCE
WITH LOADING PIN
A
0.377
0.373
32
1.75
0.875
1.1
0.9 R
0.75
1.50
1.50
1.50
6.0
0.75 DIAM REFERENCE 0.005 IN. MAX
CLEARANCE WITH LOADING PIN
A
C = B ± 0.005
1.005
0.995
0.705
0.695
0.875
1.75
3/16 RAD.
30° ± 3°
1.0
B = 2.00
1.0
6.0

A SURFACES TRUE TO CENTERLINE WITHIN 0.001 IN.; NOTCH ROOT RADIUS 0.0007 IN. MAX.; UNLESS OTHERWISE NOTED, DIMENSIONS MAY VARY ± 0.01 IN.

FIG. 2.—Sharp-Edge Notch and Smooth Tension Specimens.

mens, machining for both smooth and notch specimens was confined to the sheet edges since pressure vessels are not normally subject to surface machining. Weld specimens had their weld bead machined flush with the adjacent sheet. This does not coincide with commercial practice in the fabrication of component hardware but was necessary to determine the properties of the weldment itself.

Details of the room-temperature testing procedure follow ASTM recommendations (6). Tests at low temperature employed essentially the same techniques except that the specimen was surrounded by an insulated can containing a cold fluid. For tests at −423 F liquid hydrogen was used, at −320 F liquid nitrogen, and at −110 F a mixture of dry ice and ethyl alcohol.

Data Presentation:

While Table 2 gives all conditions investigated, not all of the corresponding data are reported in this paper, but rather an attempt has been made to illustrate typical behaviors.

In representing sharp notch data both the conventional notch strength and the nominal fracture toughness, K_{cn} are shown. The K_{cn} values were calculated by methods outlined previously (6) with the exception that the crack length at unstable fracture has been assumed equal to the original total notch length (that is, $2a = 0.30$ in.). The resulting nominal fracture toughness is a minimum value that is always less than the true value if slow crack extension occurs. As pointed out by Srawley (7), the use of nominal values has some merit since they lead to a more conservative evaluation of the alloy than do the true values. This conservatism is desirable because there is no known way of predicting the slow growth of a crack in a component when operating loads are applied. The per cent shear has also been reported in a few cases. This was determined from measurements made at or near the longitudinal centerline of the specimen.

INFLUENCE OF TEST TEMPERATURE AND SHEET THICKNESS

The behavior of titanium alloys at low temperatures has been summarized by Holden et al (8) who point out that low-temperature brittleness is increased by a high content of the interstitial elements carbon, oxygen, nitrogen, and hydrogen. For this reason most specifications limit the interstitial element content of titanium alloys to values presumably low enough to avoid significant embrittlement. However, in a previous NASA investigation (3) it was shown that the −423 F fracture toughness of 5Al-2.5Sn titanium alloy sheet could be significantly improved by further reducing the content of interstitial elements below that normally specified for commercial grade alloy. The corresponding loss in smooth tensile strength was relatively small and −423 F yield strength to density ratios well above 1,200,000 were achieved. Several other investigators, notably Schwartzberg and Keys (9) and Christian et al (10), have recently published sheet tensile data showing the influence of interstitials on the cryogenic notch properties. These investigations were made using mildly notched specimens and the results are therefore not directly comparable with those obtained in previous NASA investigations. However, the mild-notch data show a definite increase in notch sensitivity as the interstitial content is raised. Christian also presents a limited amount of data on the influence of iron which indicate this element may also have an embrittling effect.

No data exist concerning the effects of thickness on the fracture characteristics of titanium alloys at low temperature. However, a thickness effect might be expected from the behavior of high-strength alloys tested at room temperature (11,12). Thus, at room temperature the range of very low thicknesses is characterized by full shear fractures and increasing fracture toughness with increasing thickness. This behavior is apparently related to the fact that the crack tip plastic zone for a full shear fracture tends to extend an amount proportional to the sheet thickness. As the thickness further increases the fracture toughness passes through a maximum and decreases rather rapidly in a narrow range of thickness and eventually reaches the value characteristic of plane strain fracture. This transition behavior in terms of thickness has been described in detail elsewhere (11,12) and is associated with rapidly developing lateral constraint as the thickness begins to exceed about twice the crack tip plastic zone radius.

The following section will present results illustrating how the effect of test temperature on the smooth and sharp notch properties is modified by the interstitial level. In addition, the effects of thickness on the fracture properties at three interstitial levels will be shown for tests at −423 F.

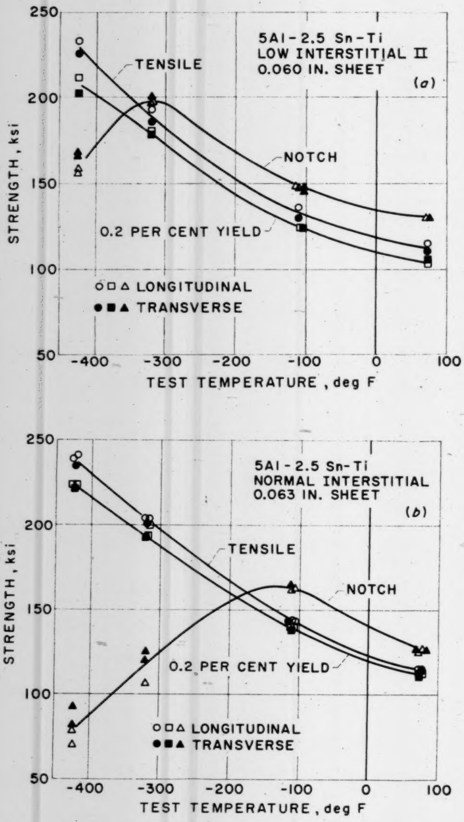


FIG. 3.—Influence of Test Temperature on Smooth and Sharp-Edge Notch Strength of (*a*) low- and (*b*) normal-interstitial 5Al-2.5Sn Titanium Alloy Sheet.

Effect of Test Temperature:

Typical examples of the influence of test temperature on the smooth and sharp-edge notch strength of a low and normal interstitial alloy are shown in Figs. 3(*a*) and (*b*) respectively for $\frac{1}{16}$-in. thick sheet. The smooth strength values at both interstitial levels exhibit the pronounced temperature dependence characteristic of titanium alloys. As might be expected, the smooth tensile and yield strengths of the normal-interstitial material are somewhat higher than those for the low-interstitial alloy. This difference, however, is less than 10 per cent over the temperature range investigated. For both alloys no practical significant difference was observed in smooth strength values between longitudinal and transverse tests.

In contrast to the behavior of smooth specimens, the sharp-notch strength trend with test temperature is different for the two interstitial levels. Thus, a rapid decrease (transition) in notch strength with decreasing temperature is observed for the low-interstitial alloy below −320 F, while this transition occurs at a much higher temperature (approximately −150 F) for the normal-interstitial sheet. Within the range of thickness investigated the notch strength transition temperature increases with increasing thickness for the normal-interstitial alloy but appears to be essentially independent of thickness for the low-interstitial material.

Influence of Sheet Thickness:

The influence of sheet thickness on the fracture characteristics at −423 F is shown in Fig. 4 for material at three interstitial levels. Smooth strength values for a given composition were essentially independent of thickness. The yield strength levels for low interstitial I, low interstitial II, and normal interstitial sheet were 202, 212, and 230 ksi respectively.

The general effect of thickness conforms to that previously observed in room-temperature tests in that the notch to yield strength ratio and the nominal fracture toughness first increase with an increase in thickness to a maximum value and then exhibit a decrease in a rather narrow thickness range. A corresponding transition in fracture appearance (per cent shear) with increasing thickness occurs and this is more pronounced than that observed in fracture toughness.

It will be noted that the magnitude of the thickness effect is considerably influenced by the interstitial element content. It would appear that the transition thickness is shifted downward with an increase in interstitials. Furthermore, at thicknesses above the transition range all three fracture parameters shown in Fig. 4 are lowered with increasing interstitial element content. Particular attention should be given to the fracture appearance curve for normal-interstitial sheet which shows a rapidly developing embrittlement between 0.050- and 0.125-in. thickness. In fact, the 0.125-in. sheet exhibits a nearly flat fracture, and it might be expected that crack propagation in sheet of this thickness and greater would be controlled by the plane strain toughness.

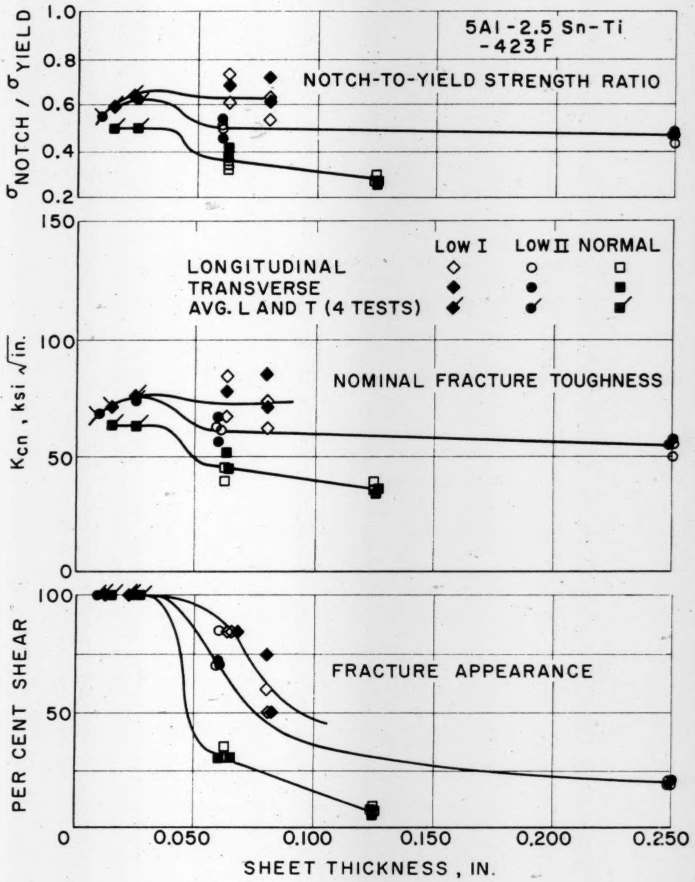


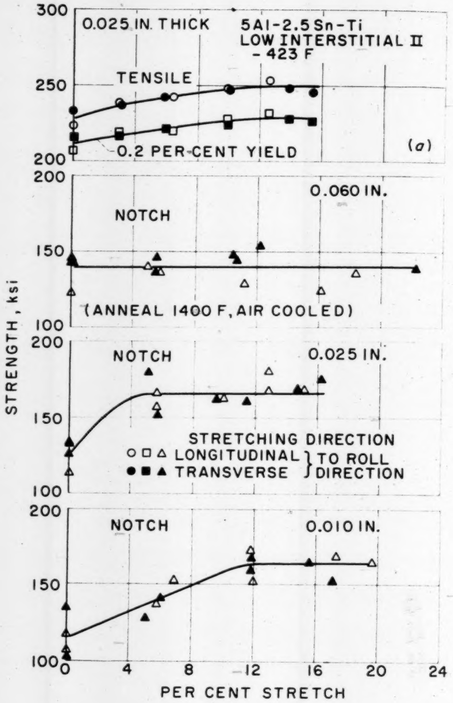
Fig. 4.—Influence of Sheet Thickness on the −423 F Sharp Notch Fracture Characteristics of 5Al-2.5Sn Titanium Alloy at Three Interstitial Levels.

INFLUENCE OF STRETCHING AND ROLLING

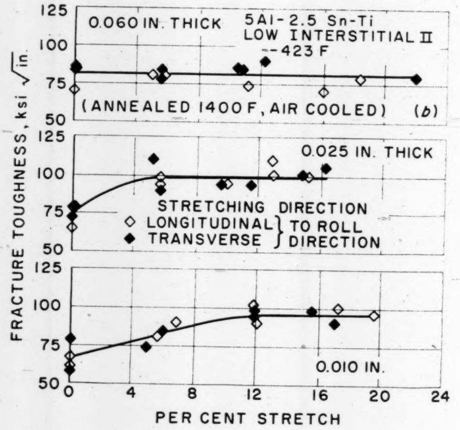
Titanium sheet alloys are commonly subjected to stretching in various fabrication procedures. However, with the exception of flattening passes sometimes given after final annealing, cold rolling of titanium alloys is not a normal practice.

There are many data (13) showing the influence of moderate stretching (5 to 10 per cent) on the room-temperature tensile properties of titanium alloy sheet. For alpha alloys and annealed $\alpha + \beta$ alloys the tensile yield strength is increased only a small amount by the first few per cent stretching and then does not change with further stretching. Geil and Carwile (14) report that room-temperature prestretching (up to 12 per cent) of cylindrical specimens did not affect the −320 F smooth or mild-notch strength of pure titanium but markedly reduced the −320 F notch strength of 4Al-4Mn titanium without a corresponding increase in smooth strength. In a study of 0.41N titanium, Ripling (15) found an improvement in room-temperature ductility of sharply notched bars with prestrains up to 6 per cent but an embrittling effect at higher prestrains. Recently Christian et al (10) reported that strengthening with no accompanying mild-notch embrittlement at −423 F is associated with double radius stretch forming of 5Al-2.5Sn titanium alloy sheet. However, no experimental details were provided.

Petunina and Poplavskaya (16) investigated the influence of cold rolling up to 30 per cent on the room-temperature tensile and bend properties of a 3Al-2V titanium sheet alloy. Reductions up to about 20 per cent produced significant increases (approximately 20 per cent) in tensile yield strength, but rapid loss in elongation, reduction of area, and bend angle. The data for 30 per cent



(a) Influence on smooth and sharp-notch strength.



(b) Influence on fracture toughness.

FIG. 5.—Influence of Stretching on −423 F Smooth and Sharp Notch Strengths and Fracture Toughness of Low-Interstitial 5Al-2.5Sn Titanium Sheet Alloy; Testing Direction Coincides with Stretching Direction.

reduction indicate a decrease in the rate of change of these properties for rolling beyond about 20 per cent.

Christian et al (10) report similar behavior for 5Al-2.5Sn titanium sheet rolled up to 50 per cent. Reductions up to 10 per cent produce an increase in strength of about 20 per cent at room temperature and 10 per cent at −423 F. Heavier rolling produced no further significant strength increase at either temperature. Mild-notch data indicated that two heats exhibited brittleness at −423 F for reductions in excess of 10 per cent. A third heat with lower interstitial content showed no embrittlement for reductions up to 20 per cent.

From the above review of published results it would be expected that moderate amounts of stretching or rolling would not produce large strength increases in alpha titanium alloys at cryogenic temperatures and that the effect of these variables on the fracture properties would depend in large part on the composition. The present study was designed to explore the influence of stretching up to a value higher than normally encountered during fabrication and to determine the effects of small reductions by rolling on the −423 F smooth and sharp-notch properties of low-interstitial sheet.

Results for Stretching:

The results for three initial thicknesses of low-interstitial sheet tested at −423 F are given in Fig. 5 as a function of per cent stretching. It is important to note that the solid and open points do not refer to longitudinal and transverse in the sense used in other figures but refer to the direction of stretching with respect to the original sheet rolling direction. All specimens were tested in the stretching direction (see Appendix). The smooth strength (Fig. 5(*a*)) is shown only for 0.025-in. thick sheet, since the other thicknesses investigated exhibited essentially identical trends. The tensile and yield strengths are increased about 10 per cent by stretching 12 per cent. Additional stretching would not appear to produce a further increase in strength.

The influence of stretching on the notch strength (Fig. 5(*a*)) and fracture toughness (Fig. 5(*b*)) depends on the initial thickness. Thus the 0.060-in. thick sheet[4] is unaffected by stretching while both the 0.025- and 0.010-in. thick sheet exhibit a definite improvement in fracture properties in the range of small stretching strains. The authors have no explanation for this effect since, as will be mentioned later, no improvement in nominal fracture toughness is noted due to prestraining equivalent amounts by cold rolling.

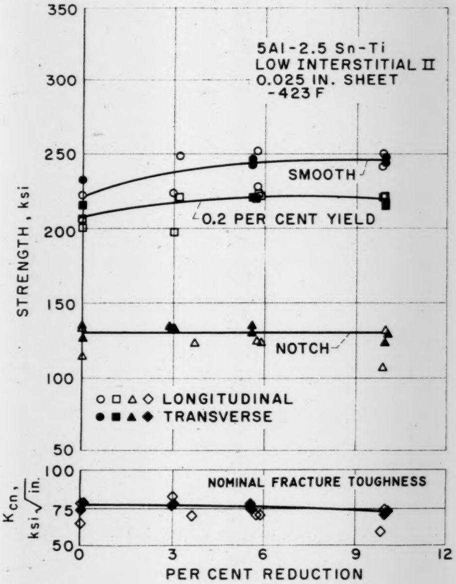


FIG. 6.—Influence of Cold Rolling on −423 F Smooth and Sharp Notch Properties of Low-Interstitial 5Al-2.5Sn Titanium Alloy Sheet.

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[4] This thickness sheet was mill line annealed at 1400 F and air cooled. The 0.025- and 0.010-in. sheet were reannelaed at NASA in accordance with Table 1.

Results for Rolling:

The results for 0.025-in. low-interstitial sheet tested at −423 F are shown in Fig. 6 as a function of reduction by rolling. Essentially identical behavior was observed for 0.010-in. thick sheet. The tensile and yield strengths increase with per cent reduction and reach a constant value between 6 and 10 per cent reduction. The elevation in tensile strength is approximately 10 per cent while the yield strength increases 7 per cent. The notch strength and fracture toughness are unaffected over the range of rolling reductions investigated.

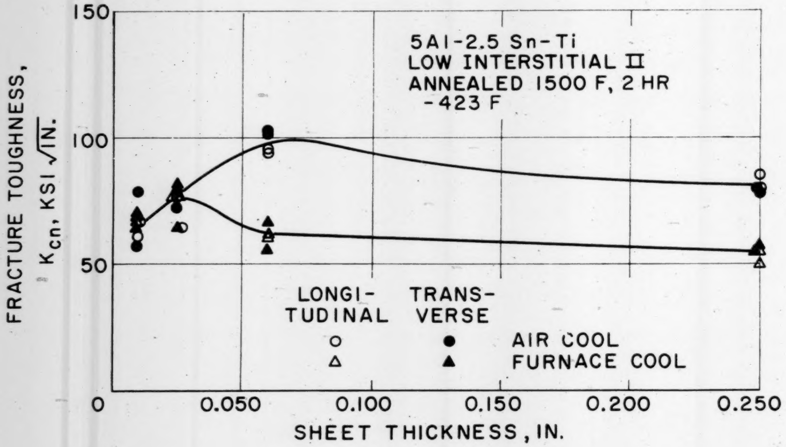

FIG. 7.—Nominal Fracture Toughness as a Function of Sheet Thickness for 5Al–2.5Sn Titanium Low-Interstitial Sheet Air Cooled and Furnace Cooled from Annealing Temperature.

ANNEALING TREATMENT

The occurrence of ordering reactions in the titanium-aluminum system is well known. Maykuth et al (17) have summarized a number of investigations of the Ti-Al binary diagram and show the presence of an ordered α_2 (assumed to be Ti_3Al) phase field in the range of low aluminum content. The lower composition boundary of this phase at 1022 F is fixed by the data of Crossley and Carew (18) at 6 weight per cent aluminum or lower.[5] It might be expected that the presence of an ordered α_2 phase could adversely affect the fracture properties of titanium alloys. Thus, data developed by Titanium Metals Corp. of America (19) show the fracture toughness of mill annealed (furnace cooled) 8Al-1Mo-1V titanium sheet to be increased about 75 per cent by a very short reanneal followed by air cooling. This increase in fracture toughness is likely due to the absence of the ordered phase in the air-cooled material.

This portion of the present investigation was designed to determine whether or not the cooling rate from the annealing temperature would affect the cryogenic fracture properties of 5Al-2.5Sn titanium alloy sheet. Specimens of various thicknesses of low interstitial II sheet were air cooled from the annealing temperature. This was accomplished by removing the sealed argon-filled specimen container from the furnace and allowing it to cool in still air. The fracture properties of air-cooled specimens are compared in Fig. 7 with those of specimens given the usual furnace cool. This latter cooling rate corresponds to that which might be encountered in a mill vacuum anneal. The smooth tensile properties were essentially identical for the two cooling rates. However, the sharp-notch behavior was quite different in the range of thicker sheet. Thus the nominal fracture toughness of sheet equal to or greater than 0.060 in. is increased about 50 per cent by air cooling.

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[5] A recent study by Clark et al (**20**) has modified this diagram for compositions greater than 7.8 weight per cent aluminum at temperatures of 1200 F and above. However, these modifications do not help establish the α_2 boundary at temperatures below 1000 F.

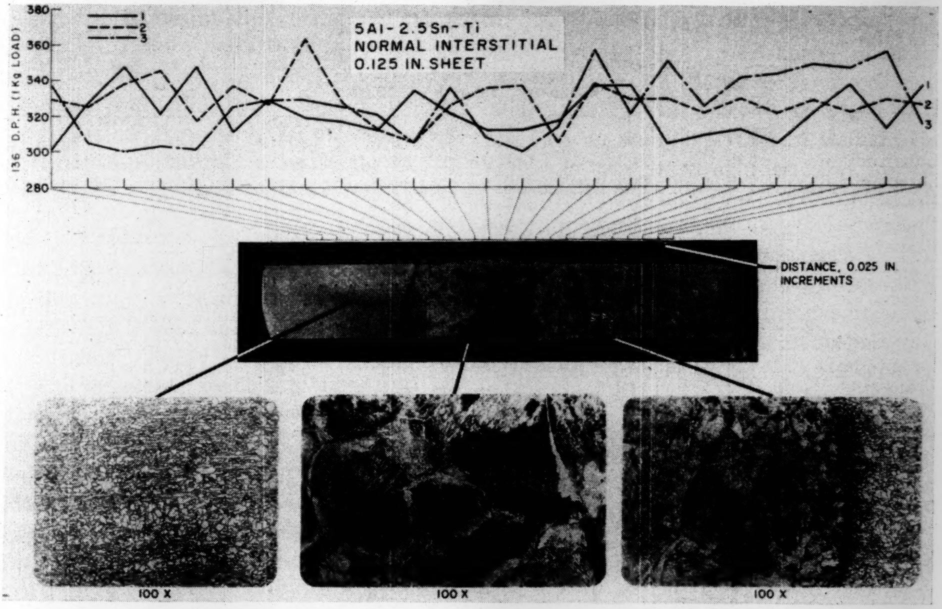

Fig. 8.—Weld Hardness Survey and Microstructure of TIG Welded Normal Interstitial 0.125-in Sheet. Etch: 30 Lactic Acid, 10 HNO_3, 5 HF.

Welding

There is apparently no published information concerning the behavior of alpha-titanium weldments in the presence of sharp notches. In the present investigation, sharp notch properties were determined for welded 5Al-2.5Sn titanium alloy sheet at two interstitial levels. Possible areas of embrittlement due to welding were first explored through careful metallographic examination of the fusion and heat affected zones and Vickers hardness surveys of a section through the weld and perpendicular to the sheet surface. Results typical for all sheet are illustrated in Fig. 8 which shows the hardness distribution along several locations through the thickness as well as the microstructure of the welded sheet. The hardness survey indicates only random variations from the base metal to the weld centerline. Examination of the microstructure reveals very large acicular grains in the weld fusion zone as the only identifiable region of possible embrittlement. Thus on the basis of these observations the notch was located at the weld centerline.

The smooth and sharp-notch properties of welded specimens at room temperature and −423 F are shown in Table

TABLE 3.—SMOOTH AND SHARP-EDGE NOTCH PROPERTIES OF BASE METAL AND WELDS IN LOW AND NORMAL INTERSTITIAL 5Al-2.5Sn TITANIUM SHEET ALLOY.

	Base Metal				Welded Metal		
Direction	Tensile Strength, ksi	0.2 Per Cent Yield Strength, ksi	Sharp-Notch Strength, ksi	Fracture Toughness, K_cn, ksi $\sqrt{\text{in.}}$	Tensile Strength, ksi	Sharp-Notch Strength, ksi	Fracture Toughness,[a] K_cn, ksi $\sqrt{\text{in.}}$
0.025-in., Low I, Room Temperature							
Longitudinal...	100.0	97.0	119.0	...	96.0	109.0	...
Longitudinal...	97.8	94.7	...	...	93.3	107.0	...
Transverse.....	99.2	92.4	118.0	...	...	108.0	...
Transverse.....	...	...	117.0	...	...	108.0	...
0.025-in., Low I, −423 F							
Longitudinal...	221.0	203.0	120.7	70.3	206.2	137.0	81.5
Longitudinal...	222.0	202.5	141.0	83.1	204.2	107.0	60.7
Longitudinal...	218.4	205.9	129.8	74.8	...	151.0	91.7
Transverse.....	212.0	208.0	141.9	83.6	...	135.0	79.3
Transverse.....	227.3	204.2	115.8	66.1	...	...	...
Transverse.....	...	...	119.6	68.0	...	...	...
0.025-in., Normal, Room Temperature							
Longitudinal...	114.4	110.0	122.8	...	112.8	125.8	...
Longitudinal...	114.3	111.0	129.5	...	112.0	128.4	...
Transverse.....	117.5	112.8	127.0	...	...	128.7	...
Transverse.....	...	...	128.7	...	...	...	...
0.025-in., Normal, −423 F							
Longitudinal...	235.0	207.0	119.0	67.1	234.0	117.2	66.1
Longitudinal...	233.0	225.0	115.2	65.2	239.3	95.1	52.8
Longitudinal...	238.0	230.5	110.3	57.2	...	111.3	62.6
Transverse.....	229.0	226.0	111.0	62.4	...	...	...
Transverse.....	235.3	224.0	99.0	60.3	...	...	...
Transverse.....	...	...	128.3	73.4	...	...	...
0.125-in., Normal, −423 F							
Longitudinal...	251.0	246.0	65.0	35.4	247.0	83.7	46.1
Longitudinal...	252.0	246.0	71.3	38.9	246.0	99.5	55.3
Transverse.....	232.0	232.0	63.4	34.5	...	89.3	49.3
Transverse.....	...	...	66.5	36.2	...	...	...

[a] For this calculation the yield strength of weld metal was taken as 95 per cent of its tensile strength.

3 for 0.025-in. normal- and low-interstitial sheet as well as the −423 F properties for 0.125-in. normal-interstitial material. In addition, the corresponding properties of the base metal are given for the purpose of comparison. It should be noted that the designations "longitudinal" and "transverse" can properly be applied only to the base metal properties since the weld metal should be essentially nondirectional.

According to Table 3 there is very little difference between the smooth and notch properties of weld metal and that of the parent plate. Thus, the higher smooth strength and lower toughness of the normal as compared with low-interstitial sheet is reflected in welds made in these alloys.

PRACTICAL SIGNIFICANCE OF RESULTS

The previous sections have described the effects of certain material production and fabrication variables on the −423 F smooth and sharp-notch properties of 5Al-2.5Sn titanium sheet alloy. No claim is made to have defined or investigated all important variables in either category. However, on the basis of the results presented it is possible to make certain observations of practical interest to the producers and users of titanium alloys.

Alloy Production Variables:

Alloy production variables included in this study were sheet thickness, interstitial level, cold rolling, and annealing treatment.

The effects of sheet thickness and interstitial level on the fracture toughness are interrelated. Thus the thickness range in which the toughness exhibits a transition (see Fig. 4) is shifted to thicker gages by a reduction in interstitial content. This behavior indicates that the need for low-interstitial material becomes more critical as the section size increases. It is possible that additional toughness in thick sheet and in heavier sections can be gained by further reductions in the interstitial content below that designated as "low" by present commercial standards. This observation is based on the behavior of only two heats (compare low I and low II in Fig. 4) but recognizes the fact that interstitial elements in extremely small quantities can influence the fracture characteristics. The small loss in smooth tensile and yield strength at cryogenic temperatures caused by reductions in the interstitials are far outweighed by the increase in crack propagation resistance.

It is the authors' opinion that further progress in reducing the interstitial (or other impurity) embrittlement of titanium alloys cannot be made without systematically studying the individual effects of the various elements.[6] Such studies would permit definition of a reasonable goal in terms of desired properties and furnish some indication of the feasibility of achieving this goal in commercial production.

The cold rolling results are very encouraging in that small amounts of reduction (up to 10 per cent) produce a slight increase in smooth strength with no loss in nominal fracture toughness. Thus it may be concluded that normal roll flattening operations after final annealing would have no deleterious effect. Other investigators have shown that substantial increases in room-temperature strength are possible with higher reductions. However, there are no data available concerning associated changes in the fracture properties. It should be noted that in a previous publication (3) by one of the present authors, data were presented for 5Al-2.5Sn titanium sheet rolled 19 per cent and tested at −423 F. The sharp-notch strength of this alloy was reduced below that of the annealed material and on this basis it was recommended that alpha titanium not be cold rolled. Reexamination of the cold-rolled specimens and sheet stock indicated surface cracking, judged to be of sufficient magnitude to explain the deleterious effects of rolling. Surface cracking is difficult to avoid in cold rolling this alloy and particular care must be used to ensure complete recrystallization during intermediate anneals.

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[6] Ogden and Jaffee (**21**) proposed an oxygen equivalent effect for the influence of interstitials on the tensile properties of pure titanium, and Schwartzberg and Keys (**9**) have applied this to representations of notch data for 5Al-2.5Sn titanium at cryogenic temperatures. Unfortunately the variations in interstitial levels for any one notch specimen type are not sufficient to establish the usefulness of this relation. It is also to be noted that Klier and Feola (**22**) attempted to establish the independent effects of the interstitials on 5Al-2.5Sn titanium bar stock using sharply notched round bars. However, due to various experimental difficulties the data were not suited for the intended purpose.

The results of the annealing treatment study indicate that the ordered α_2 phase may exist in the 5Al-2.5Sn titanium alloy at room temperature. Substantial increases in the nominal fracture toughness at −423 F apparently can be obtained by air cooling rather than furnace cooling from the annealing temperature.

Fabrication Variables:

The two fabrication variables studied were uniaxial stretching and fusion welding without filler. Stretching (up to 16 per cent) increased the −423 F smooth strength slightly and had no detrimental effect on the nominal fracture toughness of low-interstitial sheet. In fact, the toughness of thin gages appears to be improved by small amounts of stretching. The data obtained in this investigation related to tests in the stretching direction; however, the lack of directionality due to rolling would suggest that the stretched alloy is also nondirectional. Results obtained by one other investigator at −320 F on bar stock indicate that stretching may embrittle high-interstitial alpha alloys. This is another argument for minimizing the interstitial level.

Tungsten inert gas fusion welds made without filler in either low- or normal-interstitial sheet had essentially the same smooth and sharp-notch properties as the parent sheet at room temperature and −423 F. The extremely large grain size and the presence of an acicular microstructure in the weld metal have apparently no adverse effect on its toughness.

Acknowledgments:

The authors wish to express their appreciation to R. J. Kaiser and J. J. Priebe for assisting with the tests and to Anne Kendra and Frank M. Terepka for preparation of metallographic specimens and hardness surveys. The experimental heat of low-interstitial titanium sheet was furnished by Republic Steel Corp., Central Alloy District, and we are grateful to Mr. H. O. Mattes of that organization for his cooperation in the initial phases of the program. Appreciation is also due to General Dynamics/Astronautics, Inc. and to A. Hurlich, Chief of the General Dynamics/Astronautics Materials Research Group, for furnishing sheet from the commercial heat of low-interstitial material and for the fusion welding.

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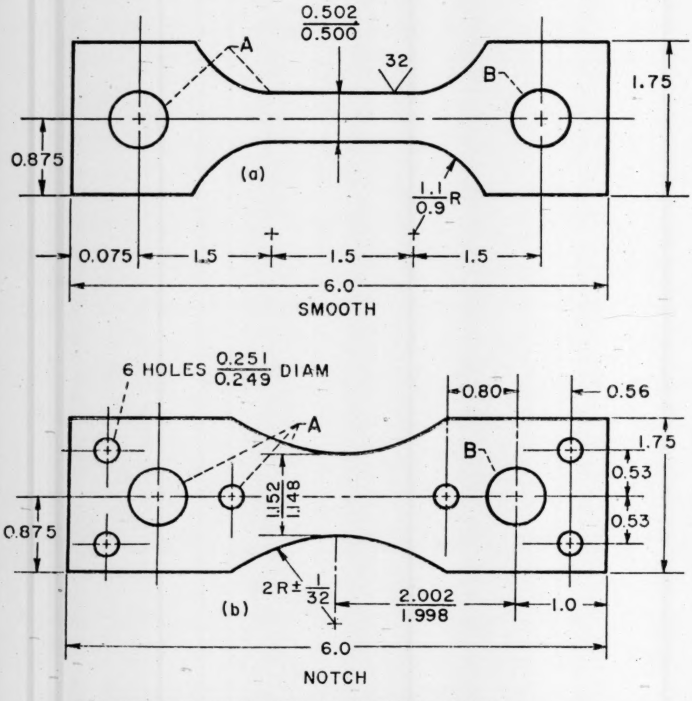
(See Appendix on page 824, and Discussion on page 825.)

APPENDIX

PROCEDURES USED IN STRETCHING, ROLLING, AND WELDING

The following describes the procedures used for room-temperature stretching, rolling, and welding of annealed sheet.


0.502
0.500
A
32
B
1.75
0.875
(a)
1.1
0.9 R
0.075
1.5
1.5
1.5
6.0
SMOOTH
6 HOLES 0.251/0.249 DIAM
0.80
0.56
A
B
1.75
0.53
0.53
1.152
1.148
0.875
2R ± 1/32
(b)
2.002
1.998
1.0
6.0
NOTCH

A SURFACES TRUE TO CENTERLINE WITHIN 0.001 IN.
B : 0.625 DIAM. REF. 0.005 IN. MAX. CLEARANCE WITH LOADING PIN
ALL DIMENSIONS ARE IN INCHES.

FIG. 9.—Smooth and Notch Stretch Specimen Blanks.

Stretching:

Stretching was performed at room temperature on the special pin-loaded stretch blanks shown in Fig. 9 having their loading axis either longitudinal or transverse to the sheet rolling direction.

The longitudinal strain distribution in the smooth stretch blanks was determined from measurements of width and thickness over the gage section of the blank before and after stretching. The strain distribution was not completely uniform. However, it should be noted that the average strain over a central 1-in. gage length was within 10 per cent of the maximum strain value.[7] The notch stretch blank was contoured so that the maximum strain occurred at the subsequent location of the notch.

After stretching, the blanks were machined to the contour of the specimens previously shown in Fig. 2.

Rolling:

Rolling was performed on individual specimen blanks at room temperature in a 4-high laboratory mill using carbide rolls 8 in. wide, and $2\frac{5}{8}$-in. in diameter with an 8 rms surface finish. Rolling speed was approximately 50 to 75 ft per min, and the rolling direction coincided with that of the as-received sheet. Care was taken to ensure uniform reduction of the blank by liberal application of lubricant before each pass, by not exceeding 3 per cent reduction per pass, and by inverting the specimen blank between successive passes. This procedure produced flat stock without camber.

Welding:

All welding was performed by the General Dynamics/Astronautics Corp. using production welding techniques. Sheets were joined by TIG fusion welds using argon gas shielding and no filler. Double pass welds (single pass each side) were made on the 0.125-in. gage material; only a single pass was required for the 0.025-in. sheet. Welds were made both parallel and perpendicular to the sheet rolling direction. Radiographs made of all welds revealed no porosity and photomicrographs showed overlap of all double pass weld beads. Specimen blanks were cut from the welded sheets with the loading axis perpendicular to the weld centerline. Weld beads were ground flush with the specimen surface.

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[7] The maximum strain was forced to occur at the transverse centerline by slightly tapering the gage section of the smooth stretching blank.

DISCUSSION

MR. JACK CHRISTIAN[1] (*presented in written form*).—The authors are to be commended for their work in an area of great importance to the design and metallurgical engineers who are using or comtemplating the use of the 5Al-2.5Sn titanium alloy for structural applications at cryogenic temperatures. Through the efforts of many investigators, it has been ascertained that very low interstitial content is desirable, if not mandatory, when employing titanium and titanium-base alloys at cryogenic temperatures, particularly at −423 F. A number of other factors may severely affect the strength and/or toughness of titanium alloys at ambient and cryogenic temperatures and thus must be investigated to improve the design of titanium structures. In addition to a number of possible studies on castings and forgings, for wrought sheet materials these include determination of the effects of processing variables such as rolling, heat treatment practice, and chemical composition (noninterstitial elements such as iron); fabrication variables, such as joining, in particular fusion welding, forming, and chemical or machine milling; and effects of service exposures such as liquid oxygen and liquid hydrogen environments, various gaseous exposures at elevated temperatures, thermal cycling, and so on, on the mechanical properties. The authors have described their work on determining the effects of several of these variables on the mechanical properties of one of the most promising titanium alloys for cryogenic service—the 5Al-2.5Sn titanium alloy.

A number of the factors studied by Messrs. Shannon and Brown have also been evaluated at General Dynamics/Astronautics and are reported in a GD/A report, *No. AR-592-1-373*, entitled "Mechanical Properties of Titanium-5Al-2.5Sn Alloy at Room and Cryogenic Temperatures," by Messrs. Christian, Hurlich, Chafey, and Watson.[2] Although some of the test specimens and procedures varied from those used in the subject investigation, the data and conclusions on the effects of chemical composition (interstitial content), test temperature (from 75 to −423 F), sheet thickness, stretching, rolling, and fusion welding on the mechanical properties of the 5Al-2.5Sn titanium alloy are in excellent agreement. A more thorough investigation on the effects of sheet thickness, stretching, and rolling (in the area of 3 to 10 per cent reduction) was performed in the study of Messrs. Shannon and Brown. In addition a very interesting study on annealing treatment was performed. It is believed that the increase of the fracture toughness of the 0.060 to 0.250-in. thick material by air cooling as compared to furnace cooling is quite significant and should be further evaluated.

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[1] Staff Scientist, General Dynamics/Astronautics, San Diego, Calif.

The authors have reported no deleterious effects on the strength or notch toughness of the 5Al-2.5Sn titanium alloy at −423 F as a result of stretching up to as much as 16 to 22 per cent (in fact, an improvement in these properties is reported). These results are similar to those found in studies at General Dynamics/Astronautics for stretch forming up to 3 to 6 per cent reduction in sheet thickness over a compound form with 30 by 60 in. radii (a part required for the aft bulkhead of a 10-ft diameter propellant tank). Both the present investigation and studies at General Dynamics/Astronautics, however, have shown that cold rolling beyond about 10 per cent reduction in area results in a decrease in the notch toughness of the 5Al-2.5Sn titanium alloy at −423 F. Although a significant difference is realized between cold rolling and stretching, it is believed that caution should be exercised when forming requires stretching or rolling beyond 10 per cent reduction if the material is to be employed for structural use at −423 F. In addition to the variables discussed above, the effects of various exposures on the mechanical properties of titanium alloys are believed to be necessary for proper selection of materials and design. In this respect General Dynamics/Astronautics is currently evaluating the effects of long-time thermal exposures and cyclic exposures in various gases (air, various partial pressures of oxygen and hydrogen gas) and at various temperatures (from −423 F to 800 F) on the mechanical properties of 0.006 and 0.013-in. thick low-interstitial 5Al-2.5Sn titanium alloy. This work is being sponsored by Materials Central, Aeronautical Systems Division of the U.S. Air Force.

MR. E. F. ERBIN[3] (*presented in written form*).—The contributions of the authors are of significant value to the development of proper titanium alloys and processing techniques for −423 F use. The influence of annealing practice in the 5Al-2.5Sn titanium alloy will be further explored as a result of this work. The innocuous effect of prestrain shown by the authors is also of value.

With respect to the influence of the beta-stabilizing element iron, Titanium Metals Corp. of America has now completed a program to evaluate its effect. Interstitial levels, oxygen, carbon, nitrogen, and hydrogen were held at levels previously determined as appropriate for −423 F use. Iron content was varied in the range 0.1 to 0.4 per cent.[3a] Sheet 0.040 in. thick was produced from each ingot and tested in the annealed condition at 70, −320, and −423 F. Smooth and notched ($K_t = 6.3$) specimens were tested at each temperature in duplicate from both the longitudinal and transverse directions of each sheet.

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[2] See p. 578.

[3] Assistant Manager, Technical Service and Development, Titanium Metals Corp., of America, New York, N. Y.

The results are shown in the accompanying Fig. 10 as the ratio of notch tensile strength to 0.2 per cent yield strength. The results indicate that above 0.2 per cent iron, a definite increase in notch sensitivity does occur.

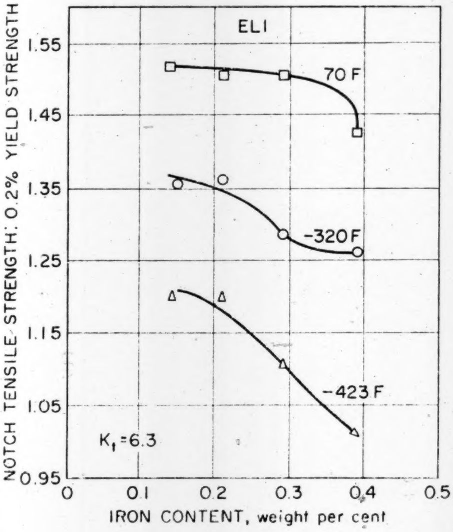

FIG. 10.—Effect of Iron in 5Al-2.5Sn ELI Grade Titanium Alloy.

NOTE.—Oxygen content of the four ingots of varying iron content ranged from 0.093 to 0.108 per cent.

The author correctly notes that reduced interstitial levels and iron content in the 5Al-2.5Sn titanium alloy results in a corresponding decrease in smooth tensile strength. This is particularly true at room temperature. A 100-sheet sample of Titanium Metals Corp. production of the 5Al-2.5Sn ELI grade titanium alloy indicates the following range of ultimate tensile strengths:

	Average Tensile Strength, psi	Range, psi
Longitudinal	111 000	106 000 to 118 000
Transverse	110 000	106 000 to 119 000

This is over 10,000 psi below that encountered in normal 5Al-2.5Sn titanium alloy.

MESSRS. K. S. JEPSON, D. CLARK, AND G. I. LEWIS[4] (*presented in written form*).—We would like to offer some comments on the hypothesis that ordering is responsible for the differences in fracture toughness of air-cooled and furnace-cooled specimens (Fig. 8 of the paper).

We feel it is unlikely that ordered α_2 could exist in a binary titanium - 5 weight per cent aluminum alloy cooled in either of the manners described. In the case of the 8, 9, and 10 weight per cent aluminum alloys used for our phase diagram investigation,[5] in order to approach equilibrium at every 50-C step from 950 to 550 C, the annealing time at all the steps in this range totaled 2000 hr; however, in order to approach equilibrium at 500 C a further 2000-hr anneal was necessary at this temperature. This extreme sluggishness of the ordering process at

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[3a] The hydrogen content of the four ingots is as follows:

Heat No.	Iron, per cent	Hydrogen, per cent
V-2420	0.14	0.0056
V-2347	0.208	0.0054
V-2348	0.289	0.0067
V-2346	0.385	0.0072

[4] Chemistry, Physics and Metallurgy Department, Royal Aircraft Establishment, Farnborough, Hampshire, England.

[5] D. Clark, K. S. Jepson, and G. I. Lewis, "A Study of the Titanium-Aluminium System up to 40 at% Aluminium," *Journal*, Inst. Metals, Vol. 91, Feb., 1963, p. 197.

low temperatures was the reason for terminating our diagram at 500 C.

Extrapolating the $\alpha/\alpha + \alpha_2$ boundary of our diagram it would appear that, if equilibrium could be achieved, ordered α_2 might form in a titanium - 5 weight per cent aluminum alloy, at about 100 C, but for the reasons outlined above it is unlikely that this equilibrium will be achieved in any reasonable period of time.

In the case of the 5Al-2.5Sn titanium alloy, it is not easy to know precisely how the addition of tin would affect the situation. This quantity of tin is approximately 1 atom per cent so that even if this were regarded as equivalent to extra aluminum it would only have a minor effect corresponding to approximately 0.5 weight per cent aluminum.

In the case of 10 weight per cent aluminum alloys it has been shown that additions of tin accelerate the kinetics of the $\alpha \rightarrow \alpha_2$ transformation[6] but it would seem unlikely that this effect would be of any significance in a titanium - 5 weight per cent aluminum alloy.

We would suggest that the process responsible for the differences observed in the fracture toughness of air-cooled and furnace-cooled 5Al-2.5Sn titanium alloy is one of short-range ordering and requires a relatively small amount of atomic movement, thus making it a more probable process at low temperatures.

MESSRS. J. L. SHANNON AND W. F. BROWN, JR. (*authors' closure by letter*).—The authors are grateful for the discussers' interest in the paper and their valuable contributions to the subject.

Mr. Christian has correctly noted a general agreement of results between the present work and his recent study of 5Al-2.5Sn titanium alloy (reference (10) of the paper). With regard to the influence of cold rolling it should be noted that the author did not investigate cold reductions greater than 10 per cent while the minimum cold reduction reported by Christian (10) was 10 per cent. Therefore the results on cold rolling effects from the two investigations are not directly comparable. However, Christian's results show that reductions greater than 10 per cent do not significantly increase the smooth strength. On the other hand, reductions greater than 20 per cent reduce the longitudinal mild notch strength while the transverse notch strength is unaffected by reductions up to 50 per cent. As mentioned in the present paper, this alloy cannot be easily cold worked to high reductions without developing surface cracks normal to the rolling direction. Perhaps cracking of this nature contributes to the reduction in longitudinal notch properties observed by the discussers at high cold reductions. On the basis of the above observations it may be concluded that cold reductions in excess of 10 per cent should not be applied to the finished annealed sheet.

Mr Erbin's systematic study of the effect of iron content on the −423 F mild notch behavior of 5Al-2.5Sn ELI grade titanium alloy is an interesting and welcome contribution to the development of this titanium alloy composition. The results in Fig. 10 indicate that iron contents in excess of approximately 0.2 per cent decrease the notch - yield strength ratio. However, it should be noted that the mild notch specimen ($K_t = 6.3$) employed gives notch-to-yield strength ratios generally well in excess of unity. Under these circumstances, it is not possible to state positively that the effects observed reflect fracture behavior and are not largely due to a modification of the plastic properties. It would be very interesting to repeat these experiments using sharply notched specimens of sufficient size to ensure that fracture behavior is being studied. In the meantime, it seems wise to limit the iron content to 0.2 per cent as suggested by Mr. Erbin's data.

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[6] Unpublished work.

Messrs. Clark, Jepson, and Lewis feel that although long range order (α_2 structure) might exist for a titanium - 5 weight per cent aluminum binary composition at about 100 C, the achievement of equilibrium at this temperature would be unlikely for the time periods involved in the annealing treatment portion of the present investigation. Additionally, they feel that the addition of 2.5 weight per cent tin does little to accelerate the $\alpha \longrightarrow \alpha_2$ transformation in the titanium - 5 weight per cent aluminum composition, even though such effects are observed in a titanium - 10 weight per cent aluminum alloy. Having no data related to these effects, the authors can neither agree nor disagree with the discussers. A systematic study of the kind and degree of order associated with a variety of annealing treatments might be of great practical value in the development of this alloy composition.

CALORIMETRY OF PORTLAND CEMENT
I. EFFECT OF VARIOUS PROCEDURES ON DETERMINATION OF HEAT OF SOLUTION

BY H. A. BERMAN[1] AND E. S. NEWMAN[1]

SYNOPSIS

Nine calculation procedures for correcting the observed temperature rise in a solution calorimeter are compared. Eight of these procedures are used in various specifications for determining the heat of hydration of hydraulic cements by the heat-of-solution method.

The best reproducibility was found with methods that use long rating periods and reading intervals rather than short ones; that use constants of stirring energy and heat leakage that are independently calculated from calibrations or other pertinent tests of the apparatus or that are obtained in part from temperatures of the initial rating period rather than from temperatures of the final rating period (provided that they are accurately obtained); and that are not restricted by definitely specified duration times for the solution and rating periods. The best accuracy was found in methods that employ solution periods longer than 20 min and in those that employ the most rigorous possible approach to the heat-leakage integration calculation consistent with the data available.

Recently there has been a revival of interest in problems associated with the determination of the heat of hydration of portland cement. Gille (**1**),[2] Piselli (**2**), Parker and Nurse (**3**), Santarelli, Piselli, and Covarelli (**4**), and Alègre (**5**) have discussed the precision of various methods and techniques and made recommendations for improvement. Brisi (**6**), Nurse and Pai (**7**), Santarelli et al (**4**), and Alègre (**5**) have been concerned with the problem of adapting the available methods to the determination of the heat of hydration of cements containing slag or pozzolan. The current Federal Specification (**8**) also includes a method proposed for use with these cements.

This discussion is generally restricted to the heat-of-solution method as applied to the determination of the heat of hydration of reasonably rapidly dissolving cements. Alègre (**5**) has pointed out that the solution method is more precise than direct hydration procedures, although it may be subject to certain systematic errors because it employs storage temperatures different from those which may be reached in field construction. Storage temperatures, however, are a matter of arbitrary choice and can be changed if desired. With the solution method it is possible, with a moderate amount of apparatus, to complete more heat-of-hydration measurements in a given time than with direct hydration methods because the samples are not stored in the calorimeter during the curing period. The instrument is in use, therefore, only for the short time required for the heat-of-solution test at each age, and the hydrating cement samples require no attention but temperature control during the curing period. Long-time tests are possible by the solution method, while the direct methods are limited to periods generally less than a month, and may require or provide time-temperature conditions of storage quite different from field conditions. Because of its precision and rapidity, the solution method is useful for acceptance testing, provided specification limits are set at a value that takes the effect of the difference between the curing temperatures and other conditions of laboratory and field into account.

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[1] National Bureau of Standards, Washington, D.C.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

The object of this paper is to review the advantages and disadvantages of the various calculation techniques used in current heat-of-solution methods, as a guide to the users of these methods, rather than to advocate the use of any one method.

CURRENT PROCEDURES

Several different solution procedures are in use at present, to be described below. In the United States there are two methods—those described in ASTM Method C 186,[3] and in the Federal Test Method Standard No. 158a, Method 3101.1 (8), which is further subdivided into three procedures. Two methods are used in Great Britain, that described in British Standard 1370: 1958 (9) and a tentative procedure for slowly dissolving cements described in Building Research Note C 548 (10). At the Centre d'Etudes et de Recherches de l'Industrie de Liants Hydrauliques (CERILH) in France, a method similar to that of B.S. 1370: 1958 is used, as recently described by Alègre (5). Santarelli et al (4) in Italy have suggested still another procedure as an improvement on existing methods. In general, the differences among these procedures may be divided into differences in (1) apparatus, (2) duration of the initial, solution, and final experimental periods, and (3) method of correction for thermal leakage and heat of stirring and evaporation.

Apparatus:

The apparatus for most of the methods consists of a cylindrical Dewar flask of approximately 0.5-liter capacity to hold the acid solution, surrounded by some type of insulation and closed with a cork stopper. Temperature changes are generally measured with a Beckmann or other differential thermometer (recorded to the nearest estimated 0.001 C) although other devices such as thermistors, thermopiles, or resistance thermometers can be used. The solution is agitated with a stirrer powered by a synchronous motor, through reducing gears if necessary. All surfaces within the calorimeter are acid resistant or are protected against hydrofluoric acid with a resistant coating. The sample is generally introduced through a funnel passing through the stopper. In the apparatus described in U. S. Bureau of Reclamation Specification 566 (11), by Santarelli et al (4), and in two types used by the Portland Cement Assn., submerged or partially submerged plastic chargers have been used designed to permit complete solution of the sample in the acid and to establish the initial temperature of the sample as that of the interior of the calorimeter at the start of the test. These last four calorimeters are submerged in a constant-temperature water bath controlled to ±0.01 C or better. The others are operated in air in a room which may or may not be automatically temperature-controlled.

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[3] Method of Test for Heat of Hydration of Portland Cement (C 186 – 55), 1961 Book of ASTM Standards, Part 4.

Duration of the Solution and Rating Periods:

The calorimetric experiment is divided into four periods chronologically—a preliminary equilibration period, an initial rating period, an intervening solution period, and a final rating period. The temperature is recorded during the last three of these.

The sample dissolves during the solution period, and the heat developed during this time is the sum of the heat due to dissolution of the sample, the net energy introduced by stirring and evaporation (hereafter called "heat of stirring"), and the heat leakage to or from the surroundings. The function of the initial and final rating periods is to determine the separate effects of stirring heat and heat leakage at times when no heat is developed in the acid solution by sample dissolution. During these periods, the rate of change of calorimeter temperature with time is a linear function of the temperature gradient between the calorimeter and the surroundings. This function may be separated into a constant term, the change due to "stirring" energy, and a variable term, the heat leakage, which is directly proportional to the temperature gradient. If the temperature change from initial to final rating period is large enough, the heat of stirring and the thermal-leakage constant can be calculated from the temperature readings taken during these periods. The initial period serves to measure the combined effects before the sample is added; the final period, after the sample is completely dissolved.

If the calorimeter temperature is

TABLE 1.—CHARACTERISTICS OF VARIOUS HEAT-OF-SOLUTION PROCEDURES FOR CEMENTS.

Method and Reference	Duration of Rating Periods, min		Duration of Solution Period, min	Reading Intervals, min	Type of Calculation^a
	Initial	Final			
ASTM Method C 186 Federal Regular (**8**)	0	20	20	20	arbitrary
Federal Alternate (**8**) ASTM Method C 186, 1949 revision	5	15	25^b	5^c	area correction, paragraph b
Federal Blended (**8**)	10	0	30	5^c	area correction, paragraph c
BS 1370:1958 (**9**)	5	7 to 8^b	12 to 17^b	1	minute-to-minute, paragraph a
BRS C 548 (**10**)	10	0	20	10 and 20	arbitrary
Nurse and Pai (**7**)	0	0	10	1	minute-to-minute, paragraph a
CERILH (**5**)	4	4	4 to 10^b	1	minute-to-minute, paragraph a
SPC (**4**)	0	0	^{b,d}	5^c	minute-to-minute, paragraph b

[a] Entries refer to section headings in text of this paper.

[b] See text.

[c] One-minute intervals during first 5 min of solution period.

[d] As determined by conformity of rate of temperature rise to normal rate without dissolution of sample, as previously determined in a separate experiment.

allowed to pass through the temperature of the surroundings (at which point there is no temperature gradient through the calorimeter walls) and the temperature change is sufficiently large, the stirring energy may be obtained as the intercept of the line on the temperature-change axis, and the heat-leakage constant may be obtained as the slope of the line. Rigorously, it is important to examine this line only on one side of the zero-gradient point because the effect of evaporation included in the "heat of stirring" is not the same for calorimeters above ambient temperature as it is for those below. For the same reason, it is better to operate a calorimeter entirely below or entirely above ambient temperature rather than to split a determination between a positive and negative temperature gradient in the hope of obtaining a resultant heat leakage of zero.

Table 1 shows the duration of the solution and rating periods and the temperature-reading intervals for the various solution methods discussed in this paper.

The identical procedures of the 1955 revision of ASTM Method C 186[3] and Federal Test Method Standard No. 158a, Method 3101.1, paragraphs 1 through 6.5 (8) (Federal Regular) employ a 20-min solution period and a 20-min final rating period. No initial rating period is used. Only three temperature readings are taken: one at the time of introducing the sample, and two more at 20-min intervals. The Federal Test Method 3101.1, Alternate procedure (paragraph 6.6) (8) (Federal Alternate), as well as the identical 1949 revision of ASTM Method C 186,[4] employs a 5-min initial rating period, a 15-min final rating period, and a solution period the length of which is determined by the time at which the rate of temperature rise becomes constant, defined as the time after which the change during each of three successive 5-min intervals does not differ from their average by more than 0.001 C. Readings are taken throughout at 5-min intervals, except for 1-min intervals for the first 5 min of the solution period. In this laboratory, a minimum solution period of 25 min is preferred with the Federal Alternate procedure, even when the constancy of rate of rise literally satisfies the specification requirements earlier. The total combined solution and final rating periods are limited to 60 min by the specification.

The British normal procedure, B.S. 1370:1958 (9) (BS 1370), employs a 5-min initial rating period, a final rating period of unspecified length (about 7 to 8 min), and a solution period the length of which is based on the attainment of constancy of the rate of temperature rise (varying from 12 to 17 min in the sample determinations available to the authors). Temperature readings are taken at 1-min intervals throughout the determination. The CERILH method (5) employs 4-min initial and final rating periods (in the example given by Alègre). The solution period is often less than 10 min (4 min in the example given), and the end of the solution period is taken as the time after which the rate of temperature change is constant to ±0.003 C per min for four consecutive 1-min intervals. As in the BS 1370 procedure (9), readings are taken at 1-min intervals throughout the determination, but a reading is also taken 30 sec after sample introduction is begun.

The Federal Test Method 3101.1, Procedure for Blended Cements (paragraph 7) (8) (Federal Blended method), designed for slowly dissolving cements, employs a 10-min initial rating period, a 30-min solution period, and no final rating period. As in the Federal Alternate procedure, readings are taken every minute during the first 5 min, and every 5 min thereafter during the solution period. The British shortened procedure (**10**), also designed for slowly dissolving materials but not officially adopted, employs a 10-min initial rating period, a 20-min solution period, and no final rating period. Only three temperature readings are taken: 10 min before and 20 min after introducing the sample, and at the time introduction is begun. Another variant on BS 1370 for slowly dissolving cements, described by Nurse and Pai (**7**), employs no initial or final rating periods and requires 1-min readings during the solution period. The constants of thermal leakage and heat of stirring are effectively determined in a separate calibration experiment. In this method the solution period is limited to 10 min.

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[4] Method of Test for Heat of Hydration of Portland Cement (C 186 – 49), 1952 Book of ASTM Standards, Part 3.

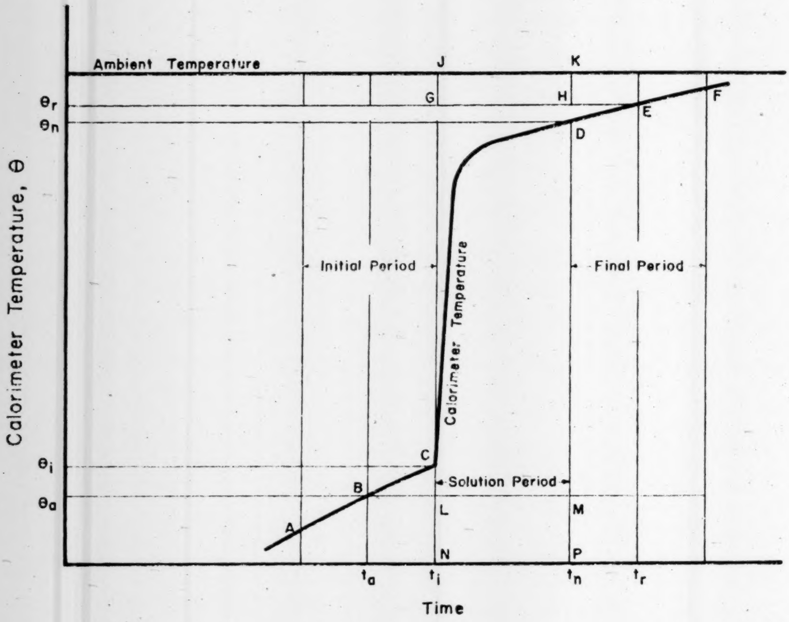

Fig. 1.—Development of Observed Temperature of a Calorimeter with Time in a Normal Determination Conducted Below Ambient Temperature.

Time and temperature intervals are not drawn to scale.

The method of Santarelli et al (**4**) (the "SPC" method), also designed for slowly dissolving materials, employs a time schedule identical to that of the Federal Alternate (**8**) and the 1949 revision of ASTM Method C 186 procedures, except that no initial or final rating periods are used. The times for starting and ending the solution period are established by the conformance of the rate of temperature rise to that obtained in a separate calibration experiment when no sample is present.

Method of Correction for Thermal Leakage and Heat of Stirring:

In all heat-of-solution methods, the observed temperature rise must be corrected to account for the net heat of stirring and evaporation and the leakage of heat to or from the surroundings. The heat of stirring is essentially constant for a constant-speed stirrer, and its constancy during a determination is assumed for computations. If uniform Newtonian heat transfer is assumed over all calorimeter boundary surfaces, the thermal leakage is proportional to the difference in temperature between the calorimeter and its surroundings and therefore varies during the course of the solution period. Based on the same assumption, the total thermal-leakage correction is, rigorously, the integral of infinitesimal corrections applied over infinitesimal time intervals and is proportional to the area bounded by *CJKDC* in Fig. 1.

This area, and the stirring energy correction, are obtained in different ways in the various correction methods although it will be shown later that all but two of the procedures are of common mathematical origin. There are three basic correction types: the area-correction method, the minute-to-minute correction method, and arbitrary correction methods.

The Area-Correction Method.—(*a*) In the area-correction method, the over-all heat-leakage correction is obtained by evaluating area *CJKDC* (Fig. 1) as the difference between two areas. The method of evaluation most closely approaching mathematical rigor uses the difference between areas *NJKPN* and *NCDPN*, each area being obtained by calculating the areas of trapezoids bounded by successive temperature readings and the time intervals between them. Accurate knowledge of ambient temperature as well as calorimeter temperature is needed at the beginning and end of each time interval. The area is multiplied by the heat-leakage constant, which may be computed from the data of the initial and final rating periods. The stirring energy is then obtained from either rating period (usually the initial) by subtracting the heat-leakage effect during that rating period from the total rate of temperature rise during the same period. The stirring energy per unit time thus calculated is multiplied by the time of the solution period to obtain the total stirring energy correction for the determination. This procedure was used in Bureau of Reclamation Specifications 566 (**11**) but is not used in any of the other acceptance test methods mentioned. It will be referred to hereafter as the bath-temperature correction method.

(*b*) Many specification methods use a variant of the preceding method in which the ambient temperature is assumed to be constant. The area *NJKPN* is then a rectangle and only one stepwise trapezoidal calculation is necessary, namely, to obtain *NCDPN*. In practice, area *CGHDC* rather than *CJKDC* is actually evaluated, as the difference between *NGHPN* and *NCDPN*. The "neglected" area, *GJKHG*, is obtained, together with the stirring energy, by applying the rate of temperature rise during the final rating period to the duration of the solution period, as will be seen in the mathematical treatment to follow. This correction procedure is used in the Federal Alternate (**8**) and the 1949 revision of ASTM C 186 methods.

(*c*) Still another variant of the area-correction method, employed in those instances when slow dissolution makes it impossible to obtain a meaningful final rating period, is the use of a previously determined value of the heat-leakage constant together with the area between the time-temperature curve *CD* and a horizontal line drawn through the average temperature of the initial rating period *BLM*. The area calculated, *LCDML*, is the difference between areas *NCDPN* and *NLMPN*. Mathematical treatment to follow shows that this area, combined with the product of the initial rate of temperature rise and the time of the solution period, correctly gives the sum of stirring energy and heat leakage. This method, assuming constant temperature surroundings and requiring only an initial rating period, is used in the Federal Blended procedure (8) and to a certain degree in the British shortened procedure (10).

The Minute-to-Minute Correction Method.—(*a*) One application of this method takes advantage of the linear relationship between the rate of temperature rise and the temperature gradient between the calorimeter and the surroundings. The rates of rise for the initial and final rating periods are plotted against the corresponding average calorimeter temperatures and a straight line is drawn between the two points obtained. This method requires the ambient temperature either to be constant or to vary linearly with time. For any calorimeter temperature, the rate of rise for the corresponding time interval may be obtained from this plot and the correction calculated. The total correction is the sum of the individual corrections over the entire solution period and includes both stirring energy and thermal leakage. This convenient method of correction is used in BS 1370 (9), Nurse and Pai's method (7), and in the CERILH method (5). The three procedures differ, however, in that the first two use the temperature at the end of each 1-min interval while the last more rigorously uses the average temperature of each interval.

(*b*) Another method which may be classified here is almost the same as described in the paragraph under "area-correction method (*a*)" except that the stirring energy and the heat-leakage constant are determined previously in calibrations rather than from the data of the determination itself. No initial or final rating periods are then needed. For each time interval of the solution period the stirring energy is known, and the heat-leakage correction may be calculated from the average temperature for the interval and the calorimeter - ambient temperature gradient. This method has been used for work in which an extraneous reaction interfered with a final rating period (12). It appears in abridged form in the SPC method (4) where 5-min intervals (except for the first 5 min) and the initial constant bath temperature are used in the calculations.

The advantage of minute-to-minute corrections is their usual simplicity and the opportunity they provide during the determination of showing exactly when the solution period is completed, or the extent to which reaction may still be continuing (12).

Arbitrary Correction Methods.—The correction procedure used in the current ASTM C 186 and Federal Regular (8) methods falls into this category. The rise during the 20-min solution period is corrected by subtracting the rise during a 20-min final rating period. This procedure assumes that the heat-leakage effect during the second period is the same as that of the first. The same assumption is made in BRS C 548 (10). Rigorously, this assumption is not true because the two temperature gradients are different. However, for rapidly dissolving materials and for flasks possessing small thermal-leakage constants, it can be shown that only a small error is involved and that results are reproducible whenever solution is complete in the 20-min period (13). The method can be extended to embrace any similar determination in which the final rating period and the solution period are equally long and will hereafter be referred to in this extended form as the 0-x-$2x$ method.

Common Mathematical Derivation of the Correction Methods:

The temperature rise due to stirring energy is constant. For an infinitesimal time interval

$$\frac{d\theta}{dt} = w \qquad (1)$$

where θ is the calorimeter temperature in degrees, t the time in minutes, and w the stirring energy in deg per min (input energy in cal per min divided by the heat capacity of the system in cal per deg).

The temperature rise due to heat leakage is proportional to the temperature gradient between the calorimeter and the surroundings. For an infinitesimal time interval

$$\frac{d\theta}{dt} = K(B - \theta) \qquad (2)$$

where B is the ambient temperature and K is the heat-leakage constant in min^{-1} (leakage in cal per min per deg divided by the heat capacity of the system in cal per deg).

The total temperature rise to be subtracted from the observed temperature rise is the sum of these two effects:

$$\frac{d\theta}{dt} = w + K(B - \theta) \qquad (3)$$

or

$$\Delta\theta = wT_x + K \int_{t_i}^{t_n} (B - \theta)\, dt \qquad (4)$$

where t_i is the time of sample introduction, t_n the end of the solution period (designated t_f by the Italian and French authors), and $T_x = t_n - t_i$.

The integral term in Eq 4 appears graphically in Fig. 1 as area *CJKDC*, where the entire determination is made below ambient temperature. If part or all of the determination is made above the ambient temperature, the equation is still valid, the proper sign changes being made automatically during the computation. Note, however, as discussed above, that errors are introduced if the determination takes place partly above and partly below the ambient temperature, because the value of w may be changed owing to the difference in evaporation rate for temperatures below and above the ambient temperature.

Another effect that is neglected in this computation is the change of the heat capacity of the solution caused by the addition of the sample. This change causes a given quantity of heat increment to affect the initial and final rating periods differently. A similar error is introduced by the change of heat capacity of the solution and sample with changes in temperature within the calorimeter. Both these errors, particularly the latter, are minimized by performing the calibration and the determination over the same temperature range, provided the calibration is made with a standard substance very close in heat capacity to that of the substance tested.

Bath-Temperature Correction Method.—The most convenient and most accurate rigorous approach to evaluating the integral term of Eq 4 is to sum up by either Simpson's or the trapezoidal rule (14) the areas formed by each temperature reading during the solution period and the time interval between successive readings. This summation is done separately for ambient and calorimeter temperatures. The difference between the two summations is area *CJKDC* in Fig. 1. Thus from Eq 4, rewritten,

$$\Delta\theta = wT_x + K \int_{t_i}^{t_n} (Bdt - \theta dt)$$

By the trapezoidal rule

$$\int_{t_i}^{t_n} B dt = \frac{B_0 + B_1}{2} + \frac{B_1 + B_2}{2} + \frac{B_2 + B_3}{2} + \cdots + \frac{B_{(n-1)} + B_n}{2}$$

where the subscripts represent the time in minutes after introduction of the sample. The summation may be written $B_x T_x$, and if B is constant may be calculated as that product.

The integral

$$\int_{t_i}^{t_n} \theta dt = \frac{\theta_0 + \theta_1}{2} + \frac{\theta_1 + \theta_2}{2} + \frac{\theta_2 + \theta_3}{2} + \cdots + \frac{\theta_{(n-1)} + \theta_n}{2}$$

or

$$\theta_0/2 + \theta_1 + \theta_2 + \cdots + \theta_{(n-1)} + \theta_n/2$$

and may be written $\theta_x T_x$. The total correction is then

$$\Delta\theta = wT_x + K(B_x T_x - \theta_x T_x) \ldots\ldots (5)$$

The value of K is found as follows:

For the initial period, subscript a,

$$(d\theta/dt)_a = V_a = w + K(B_a - \theta_a) \ldots (6)$$

From the final period, subscript r,

$$(d\theta/dt)_r = V_r = w + K(B_r - \theta_r) \ldots\ldots (7)$$

Subtracting,

$$V_a - V_r = K[(B_a - \theta_a) - (B_r - \theta_r)]$$

and

$$K = \frac{V_a - V_r}{(B_a - \theta_a) - (B_r - \theta_r)} \ldots\ldots (8)$$

The SPC Correction.—If ambient temperature is considered constant, $B = B_a = B_x = B_r$, Eq 4 may be rewritten:

$$\Delta\theta = wT_x + KBT_x - K\int_{t_i}^{t_n} \theta dt \ldots (9)$$

This equation is the same as Eq 3 of Santarelli et al (4), rewritten in the terminology used here. As in the preceding section, the integral term is calculated by stepwise summation of the calorimeter temperatures over the solution period, but using units of t = 5-min periods. However, the Italian authors use the temperature at the end of each interval for each integration step instead of the average temperature, resulting in the following rectangular rather than trapezoidal summation:

$$\int_{t_i}^{t_n} \theta dt = \frac{\theta_1 + \theta_2 + \theta_3 + \theta_4 + \theta_5}{5} + \theta_{10} + \theta_{15} + \cdots + \theta_n$$

The consequent total correction is therefore slightly smaller than if trapezoidal areas were used. K and w are determined by independent methods in the SPC correction.

Federal Alternate and ASTM Method C 186 (1949 Revision) Corrections.—If the ambient temperature is considered constant, Eq 8 becomes:

$$K = \frac{V_a - V_r}{\theta_r - \theta_a}$$

and Eq 5 becomes

$$\Delta\theta = T_x(w + KB) - K\theta_x T_x \ldots\ldots (10)$$

Adding and subtracting the term $K\theta_r T_x$,

$$\Delta\theta = T_x(w + KB - K\theta_r) + K\theta_r T_x - K\theta_x T_x$$

From Eq 7,

$$V_r = w + KB - K\theta_r$$

Therefore,

$$\Delta\theta = V_r T_x + K(\theta_r T_x - \theta_x T_x) \ldots (11)$$

As in the bath-temperature-correction method, $\theta_x T_x$ is determined by trapezoidal summation, but 5-min periods are used as the unit for T, and the summation therefore becomes

$$\theta_x T_x = \frac{\theta_0/2 + \theta_1 + \theta_2 + \theta_3 + \theta_4 + \theta_5/2}{5} + \frac{\theta_5 + \theta_{10}}{2} + \cdots + \frac{\theta_{n-5} + \theta_n}{2}$$

$\theta_x T_x$ appears graphically in Fig. 1 as area *NCDPN*. $\theta_r T_x$ is the area *NCGHPN*, and the quantity in parentheses in Eq 11 is the difference between these areas or area *CGHDC*. This calculation method is used in the Federal Alternate and ASTM C 186 (1949 revision) procedures.

Federal Blended Correction.—If Eq 5 is modified as in the preceding section but initial rating period data are used instead of final rating period data,

$$\Delta\theta = T_x(w + KB) - K\theta_x T_x$$

Adding and subtracting $K\theta_a T_x$,

$$\Delta\theta = T_x(w + KB - K\theta_a) + K\theta_a T_x - K\theta_x T_x$$

From Eq 6,

$$V_a = w + KB - K\theta_a$$

Therefore,

$$\Delta\theta = V_a T_x + K(\theta_a T_x - \theta_x T_x) \quad \ldots\ldots (12)$$

Here again, $\theta_x T_x$ is area *NCDPN* and is evaluated as for Eq 11 above. $\theta_a T_x$ is area *NLMPN*, and the expression in parentheses in Eq 12 is the difference between these areas, or area *LCDML*.

This is the calculation used in the Federal Blended Method. No final rating period is needed, but K, the heat-leakage constant, must be determined from calibrations which do include a final rating period.

The BS 1370 and CERILH Corrections.—For constant ambient temperature, Eq 3 may be rewritten

$$\frac{d\theta}{dt} = A - K\theta$$

This equation may be plotted as a straight line on rate-temperature coordinates, using a pair of points taken from the initial and final rating periods, (V_a, θ_a) and (V_r, θ_r). Individual corrections for each 1-min interval are then taken for the temperature of the interval directly from the plot. The British method uses the temperature at the end of the interval, the French method, more rigorously, the average temperature of the interval. The Nurse and Pai method is essentially the same as the BS 1370 method, but a limit of 10 min is set to the solution period. The procedures are graphical solutions of Eq 4.

The 0-x-2x Correction Procedures of the Current ASTM C 186 and Federal Regular Methods.—For constant ambient temperature, Eq 11 may be used:

$$\Delta\theta = V_r T_x + K(\theta_r T_x - \theta_x T_x) \quad \ldots\ldots (13)$$

If the time of the final rating period equals the time of the solution period, T_x, then the total temperature rise during the final period is $V_r T_x$. This is the entire correction used in the 0-*x*-2*x* method. The remainder of the expression $K(\theta_r T_x - \theta_x T_x)$ is neglected. In this sense, the method is arbitrary. The neglected portion of the correction averaged 0.011 C for dry cement and 0.004 C for the hydrated paste, or about 0.9 cal per g in the heat of hydration of 30 normal rapidly dissolving cements routinely tested in this laboratory. Since the same kind of error appears in the heats of solution of both the dry and the hydrated cement, the net error in their difference, the heat of hydration, is reduced. Thus, the arbitrary nature of the calculation does not greatly affect the results of the determination, although there may be a systematic error as large as 1 cal per g due to the usually more rapid dissolution of the hydrated sample.

The BRS Note-C 548 Correction.—The British shortened procedure also involves an arbitrary calculation based on abridgement of Eq 11 to $\Delta\theta = V_r T_x$. However, V_r is determined, not from a final rating period by observation, but by calculation from a previously determined value of K and the initial rating period. The value of K is defined as $(V_a - V_r)/(\theta_n - \theta_i)$, about 0.5 per cent greater than the value obtained by Eqs 8 or 10, and is calculated from a calibration run which includes a final rating period. Then

$$V_r = V_a - K(\theta_n - \theta_i)$$

and

$$\Delta\theta = V_r T_x = T_x[V_a - K(\theta_n - \theta_i)]$$

Since $\theta_n = \theta_{20}$, $V_a = \theta_i - \theta_{-10}$, and $T_x = 2$ (10-min periods),

$$\begin{aligned}\Delta\theta &= 2[(\theta_i - \theta_{-10}) - K(\theta_{20} - \theta_i)] \\ &= -2[K(\theta_{20} - \theta_i) - (\theta_i - \theta_{-10})]\end{aligned}$$

Of the nine calculation procedures discussed above, two contain arbitrary approximations, but are nearly equivalent mathematically to the others. The remaining seven procedures are all based on a different mathematical form of the same original equation. One of these seven procedures is more rigorous from an experimental point of view because it accounts for the temperature changes in the surroundings. Since, except for this last difference, the seven methods are of common derivation, any advantage that might arise from using a particular method is not due to the rigor of the mathematics but to the effects that normal accidental errors in the determination produce in the calculation procedure. The accuracy of the observations, the degree to which they are rounded off, the size of various correction terms, the accuracy with which the end of the solution period is chosen, the choice of time intervals for temperature readings, will all have different effects on different calculation procedures even though they are derived from the same original equation.

EXPERIMENTAL

A rapidly dissolving material, zinc oxide, was used as a convenient means of producing the temperature rise in the calorimeter encountered in a normal heat-of-solution determination on portland cement. Readings were taken every minute for 15 min before addition of the zinc oxide and for 60 min thereafter. With this procedure, the corrected rise could then be calculated by each of the methods above enumerated for solution periods of from 3 min to the maximum time permitted by the data and the method used. A comparison of the different calculation methods and of the effect of different solution periods was thus possible, from the standpoints of both precision and accuracy.

When the heat produced by solution of the sample is completely registered in the temperature rise, the corrected temperature rise should be constant thereafter from minute to minute for different choices of solution period. Precision of a calculation method, therefore, is indicated by the extent to which the corrected rise is reproduced from minute to minute in the extended calculation. Accuracy of a calculation method (for the solution period specified) is indicated by the degree to which the method produces the same result as that obtained when the solution period is definitely complete. Potential accuracy of a calculation method is indicated by comparing its eventual corrected rise (when solution is completed) with that obtained with the bath-temperature correction method (where no assumptions are made and ambient temperatures are measured and corrections made).

Additional data on the over-all reproducibility of the three Federal specification methods, which serve to evaluate the apparatus and procedure as well as the calculations, have been collected

TABLE 2.—PRECISION OF MEASURED HEAT CAPACITIES OF VARIOUS FLASK-CALORIMETER-THERMOMETER ASSEMBLIES, BY THE THREE FEDERAL CALCULATION METHODS (**8**). (Table 2 continued on next page.)

Method	Federal Alternate[a]		Federal Regular[b]		Federal Blended[c]	
Date, month and year	Heat Capacity, cal per deg	Standard Error,[d] cal per deg	Heat Capacity, cal per deg	Standard Error, cal per deg	Heat Capacity, cal per deg	Standard Error,[d] cal per deg
7-62	399.10	0.71	398.30	0.62	398.70	0.14
	397.69[e]	0.58	397.37[e]	0.49	398.01[e]	0.39
	392.42	0.02	391.82	0.19	392.76	0.62
	393.35[e]	0.52	392.26[e]	0.20	393.09[e]	0.33
12-61	398.48	0.14	397.98	0.17	398.84	0.31
	393.06	0.49	391.98	0.24	392.87	0.18
11-61	396.50	0.12	396.13	0.13	396.80	0.35
	397.48	0.24	396.16	0.09	397.39	0.60
9-61	394.41	0.32	394.27	0.32	394.29	0.31
	396.62	0.40	395.80	0.25	396.24	0.52
7-61	397.45	0.28	397.11	0.05	397.75	0.40
	395.49	0.02	395.01	0.16	396.02	0.87
3-61	398.92[f]	0.20	397.37[f]	0.23	398.88[f]	0.23
	395.79	0.65	395.45	0.61	395.45	0.85
	398.89	0.49	398.44	0.27	399.00	0.30
12-60	397.87	0.11	397.46	0.34	397.99	0.38
	394.88	0.74	394.27	0.56	394.37	1.08
8-60	395.67	0.78	395.20	0.61	396.18	0.67
	398.07	0.05	397.35	0.21	398.18	1.05
4-60	396.22	0.73	395.94	0.88	395.98	1.02
	398.11	0.26	397.58	0.26	397.52	0.62
3-60	386.50	0.12	385.86	0.24	385.97	0.21
2-60	392.72	0.22	392.60	0.16	392.35	0.37
1-60	412.78[g]	0.31	411.97[f,g]	0.41	412.08[f,g]	0.22
12-59	395.13	0.17	395.05	0.14	394.90	0.49
4-59	390.06	0.29	389.78	0.26	389.83	0.19
3-59	396.96	0.09	395.85	0.26	397.02	0.54
2-59	396.56	0.35	395.90	0.21	396.33	0.45
	393.90[h]	0.71	393.51[h]	0.71	392.99[h]	0.88
12-58	389.40	0.36	389.05	0.50	389.36	0.51
	389.70	0.73	389.33	0.29	389.84	0.05
11-58	393.61	0.25	393.21	0.24	393.67	0.17
	392.08[h]	0.21	391.51[h]	0.47	391.78[h]	0.42

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[a] Reference (**8**), Paragraph 6.6, and ASTM Method C 186, 1949 revision.

[b] Reference (**8**), Paragraph 4.4, and ASTM Method C 186, 1955 revision.

[c] Reference (**8**), Paragraph 7.2.

[d] To compare with the precision values reported by other investigators, standard error for triplicate determinations can be converted into standard deviation of a single determination by multiplying by 1.732. Standard error of triplicates can be converted into root mean square deviation by multiplying by 1.414. All values reported in this table are the results of triplicate determinations except where otherwise indicated.

[e] Determined by a new operator.

[f] Duplicate determinations.

[g] Paraffin-coated flask.

[h] Four determinations.

TABLE 2.—*Continued.*

Method	Federal Alternate[a]		Federal Regular[b]		Federal Blended[c]	
Date, month and year	Heat Capacity, cal per deg	Standard Error,[d] cal per deg	Heat Capacity, cal per deg	Standard Error, cal per deg	Heat Capacity, cal per deg	Standard Error,[d] cal per deg
10-58	396.44	0.09	395.79	0.19	396.32	0.25
	394.03	0.12	393.71	0.06	393.80	0.04
6-58	396.55	0.06	395.70	0.17	396.81	0.10
	392.19	0.66	391.85	0.57	391.90	0.59
	389.77	0.22	389.41	0.13	389.73	0.34
4-58	390.44	0.42	390.13	0.40	390.05	0.42
	393.10	0.18	392.67	0.09	393.22	0.30
	400.07[h]	1.14	399.38[h]	1.13	399.89	1.09
2-58	398.50	0.37	397.49	0.18	398.53	0.70
	392.21	0.89	392.24	0.51	392.95	0.43
1-58	393.2	0.45	392.8	0.39	393.3	0.80
	398.6	0.31	397.6	0.00	398.3	0.98
Pooled standard error for triplicates		0.48		0.44		0.60

from 45 sets of replicate calibrations (mostly triplicates) with zinc oxide made over the course of 4½ yr in two calorimeter assemblies with different flask-thermometer combinations. No results for portland cement are reported here. Consequently, the standard errors reported do not include the variations inherent in the hydration procedure or in the handling of the dry and hydrated samples; they include only the variation to be expected in the heat-of-solution determination with a rapidly dissolving material.

For these determinations, zinc oxide was prepared as specified in the current ASTM Method C 186 and in Federal Test Method Standard 158a, Method 3101.1. Apparatus meeting the Federal and ASTM Specifications was used, in which the 300-rpm geared motor is connected by rubber belts to the stirring shafts. The stirring speed was 630 rpm.

TEST RESULTS

Table 2 summarizes the series of 45 calibrations with various vacuum flask-thermometer assemblies by means of the three calculation methods of Federal Test Method 158a, Method 3101.1 (8). The table indicates the degree of precision possible with the apparatus specified in this method, when placed in a constant-temperature room controlled during a determination to ±0.2 C.

Table 3 presents the data of the extended determination with zinc oxide. Figure 2 is a comparison of the results of the various calculation methods discussed in the section entitled "Common Mathematical Derivation of the Correction Methods" applied to the determination of Table 3. The corrected rise obtained by arbitrarily ending the solution period at a given time T_x is plotted against that time for each method separately.

Figure 3 shows, for each calculation method, the deviation of the corrected rise at each value of T_x from the corresponding corrected rise in the bath-temperature correction method.

DISCUSSION

Length of the Solution Period:

The most striking observation to be made from Fig. 2 is the fact that almost all the methods require a minimum period of 20 to 25 min before the calorimeter may be said to have reached equilibrium. The most rigorous method, the bath-temperature correction method, reached a fairly constant corrected rise of 4.547 ± 0.002 C at about 22 min. Within the same tolerance, the Federal Alternate method reached a constant corrected rise of 4.546 C at 21 min, Federal Regular 4.548 C at 22 min, BS 1370 4.547 C at 25 min, BRS C 548 4.547 C at 25 min, and CERILH 4.547 C at 22 min. Exceptions are the Federal Blended method which reached a con-

TABLE 3.—OBSERVED TEMPERATURES DURING SOLUTION OF ZINC OXIDE IN VACUUM-FLASK CALORIMETER.

Time, min	Temperature			Time, min	Temperature		
	Calorimeter		Room, deg C		Calorimeter		Room, deg C
	deg B	deg C			deg B	deg C	
−15....	0.336	20.426	25.26	24....	4.986	25.076	...
−14....	0.341	20.431	25.26	25....	4.987	25.077	...
−13....	0.346	20.436	25.28	26....	4.988	25.078	25.19
−12....	0.351	20.441	...	27....	4.989	25.079	25.10
−11....	0.356	20.446	25.28	28....	4.990	25.080	25.10
−10....	0.361	20.451	25.31	29....	4.991	25.081	25.12
−9....	0.366	20.456	25.33	30....	4.992	25.082	...
−8....	0.371	20.461	25.29	31....	4.993	25.083	25.29
−7....	0.376	20.466	25.30	32....	4.994	25.084	...
−6....	0.381	20.471	25.27	33....	4.995	25.085	25.32
−5....	0.386	20.476	25.26	34....	4.996	25.086	...
−4....	0.391	20.481	25.31	35....	4.997	25.087	...
−3....	...	...	...	36....	4.999	25.089	...
−2....	0.402	20.492	25.20	37....	5.000	25.090	...
−1....	0.407	20.497	25.22	38....	5.001	25.091	25.18
0....	0.412	20.502	...	39....	5.002	25.092	25.26
+1....	4.82	24.91	...	40....	5.003	25.093	25.29
2....	4.972	25.062	...	41....	5.004	25.094	25.31
3....	4.974	25.064	25.18	42....	5.005	25.095	...
4....	4.974	25.064	25.09	43....	5.006	25.096	25.26
5....	4.974	25.064	25.09	44....	5.007	25.097	...
6....	4.975	25.065	25.09	45....	5.008	25.098	...
7....	4.975	25.065	...	46....	5.009	25.099	25.22
8....	4.975	25.065	25.09	47....	5.010	25.100	...
9....	4.975	25.065	...	48....	5.012	25.102	25.11
10....	4.975	25.065	...	49....	5.013	25.103	25.12
11....	4.976	25.066	25.22	50....	5.014	25.104	25.18
12....	4.976	25.066	25.27	51....	5.015	25.105	25.28
13....	4.977	25.067	25.22	52....	5.016	25.106	...
14....	4.978	25.068	25.19	53....	5.017	25.107	25.30
15....	4.979	25.069	25.12	54....	5.018	25.108	25.29
16....	4.979	25.069	25.11	55....	5.019	25.109	25.29
17....	4.980	25.070	...	56....	5.020	25.110	25.29
18....	4.981	25.071	25.11	57....	5.021	25.111	...
19....	4.982	25.072	25.11	58....	5.023	25.113	25.28
20....	4.984	25.074	...	59....	5.024	24.114	25.24
21....	4.984	25.074	25.19	60....	5.025	25.115	25.27
22....	4.984	25.074	25.21	61....	5.026	25.116	25.29
23....	4.985	25.075	25.28				

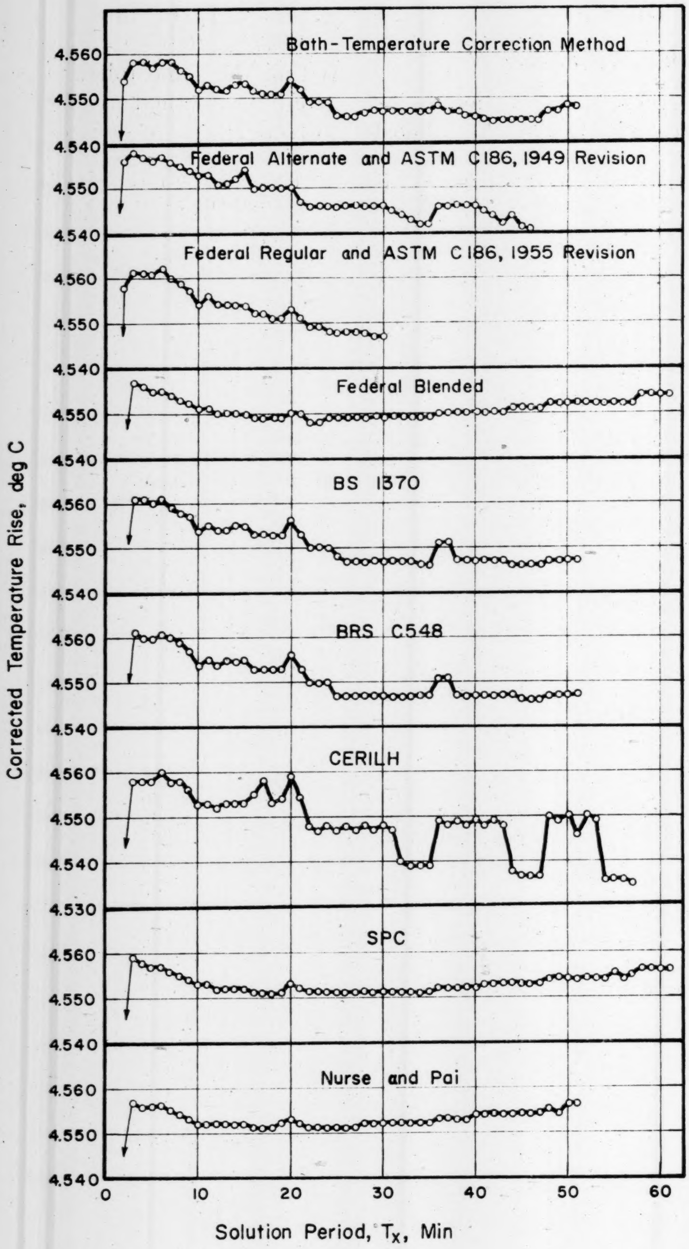


FIG. 2.—Variation of Corrected Rise with Duration of Solution Period, for the Various Calculation Methods.

stant 4.549 C at 10 min, the SPC method, 4.551 C at 10 min, and Nurse and Pai, 4.552 C at 10 min. No such rapid approach to a constant value is shown by the bath-temperature correction method, which we have taken as the most rigorous method because it takes more experimental parameters into account. It is reasonable to assume, therefore, that the slow approach to equilibrium is real, and that the three methods approach a constant value more quickly because of some sort of compensating factor in the earlier portion of the run.

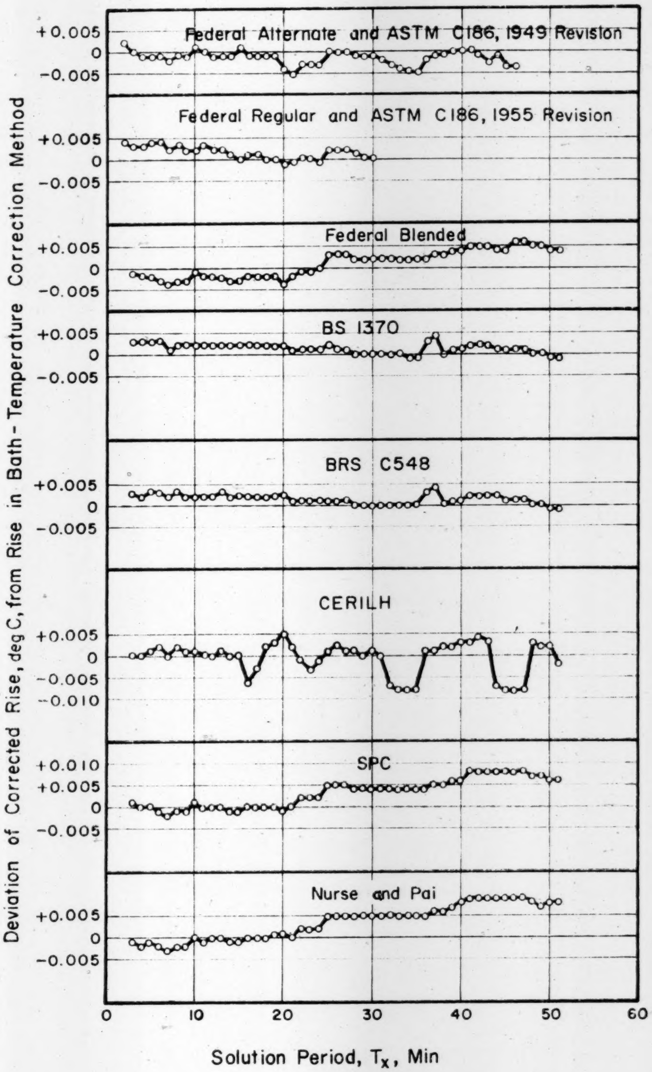


FIG. 3.—Deviation of the Corrected Rise of the Various Calculation Methods from the Corrected Rise of the Bath-Temperature Correction Method, Plotted Against Duration of the Solution Period.

It should be observed also that, by almost all the correction methods, an early plateau of constant values for the corrected rise is obtained which may be erroneously interpreted as an equilibrium if the determination is not continued beyond that point. If readings are taken 1 min apart, the calorimeter temperature will appear to have reached equilibrium, whereas later readings taken 5 min apart will show that it has not been reached. In many determinations in this laboratory, the observed temperature drops at the end of the rapid-rise period, then levels off and rises again. The authors have experienced this effect to a considerable degree when using a platinum-lined calorimeter with a separate $\frac{1}{8}$-in. thick brass cover. The phenomenon disappeared when a one-piece calorimeter with a lightweight (0.010 in.) curved upper portion was substituted **(12)**.

The pseudo-equilibrium or reverse-rise effect observed during the first 10 min is attributed to unequal distribution of the heat of solution at the beginning of the run. The heat is released in the solution near the thermometer. As heat is transferred from the solution to the cork stopper or other closure and to the glass a drop in temperature may be observed, or it may appear temporarily constant before a further rise is observed.

When the vacuum flask specified in the heat-of-hydration procedures is used, accurate values for the heat of solution cannot be expected unless the solution period is at least 25 min. It should be borne in mind, however, that if the same apparatus components are used in the determinations of the heat of solution of both the dry and hydrated cement, and the same technique of addition of the sample is used in both cases, the errors of using a shorter period may be canceled provided that the dry and hydrated cements dissolve at the same rate. The heat of hydration obtained may then be correct except for the small error introduced by shortening the time of the calibration, an error that is about one-tenth the error introduced by the same factor in the heat-of-solution determinations.

For those methods in which the duration of the solution period is specified or specific criteria are given for determining the cutoff time, the error due to shortening the solution period may be estimated by comparing the corrected rise at equilibrium with that calculated strictly according to specification. In the determination shown in Table 3, the errors shown in Table 4 would have been obtained.

The Federal Regular and BRS C 548 methods suffer in this comparison because in almost all the methods the 20-min corrected rise is erratic due to an abrupt change in the thermometer reading at that time. If the results at 18 and

TABLE 4.—POSSIBLE ERRORS THAT WOULD FOLLOW SHORTENING OF SOLUTION PERIOD.

	Specification Rise	Equilibrium Rise	Difference
Federal Alternate:			
(This laboratory)	4.546 at 25 min	4.546	0.000
(Literal)	4.553 at 10 min	4.546	0.007
Federal Regular	4.553 at 20 min	4.548	0.005
Federal Blended	4.549 at 30 min	4.549	0.000
BRS C 548	4.556 at 20 min	4.547	0.009
CERILH	4.558 at 4 min	4.548	0.010
Nurse and Pai	4.552 at 10 min	4.552	0.000

22 min are averaged, the error due to shortening the solution period to 20 min would be 0.003 and 0.006 C respectively. This effect serves to illustrate the danger of relying on single thermometer readings rather than on a series of readings, or of specifying exactly the duration of the solution period.

Any of these methods can be made more dependable by continuing the solution period as long as is necessary to attain equilibrium.

Minute-to-Minute Reproducibility:

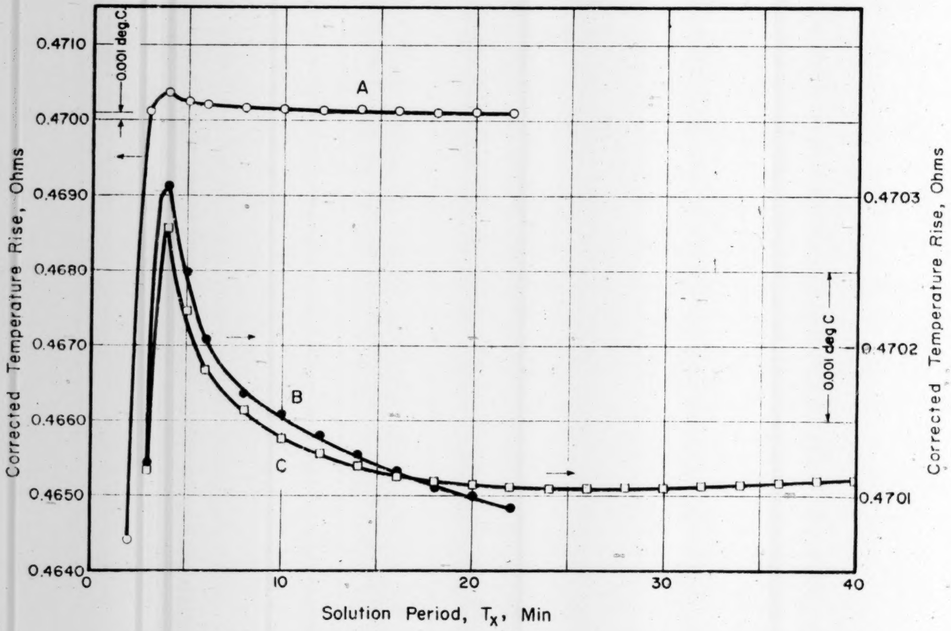
Figure 2 also illustrates the tendency of a particular calculation method to produce the same answer regardless of when the solution period is ended. Failure to do so after equilibrium is reached is due to variation in thermometer readings, which may be caused either by temperature lags within the calorimeter or by the limited sensitivity of the Beckmann scale. The scale limitation results occasionally in apparently abrupt changes in thermometer readings. These changes occur because the temperature rise for each minute may be slightly greater than 0.001 C or an integral number of thousandths of a degree. After several readings have been taken, the accumulating fractional differences produce an additional change that is discernible and is read by the operator as a rise 0.001 C greater than the temperature increases for the preceding minutes.

When the rate of temperature rise during a rating period is measured from readings only a few minutes apart, the error due to abrupt variations in thermometer readings is greater than it is when readings are taken many minutes apart. For this reason, the CERILH method shows sudden changes in the corrected rise at $T_x = 32, 36, 43$, and 48, corresponding to changes in observed temperatures at $T_x = 36$ and 48. BS 1370 and BRS C 548 show the same effect at $T_x = 36$ and 38, Federal Alternate at 30 to 34 and 36.

There is no such abrupt change in the bath-temperature correction, Federal Blended, or SPC methods. These methods do not use Eq 11 or variants thereof which contain the expression V_r, the final rate of temperature rise. The value of V_r changes with abrupt changes in thermometer readings and may therefore introduce sudden changes in the calculated corrected rise. The Federal Blended method uses V_a and a constant value of K (Eq 12), and the SPC method uses constant values of K and w (Eq 9), all of which quantities remain the same regardless of the duration of the solution period. The bath-temperature correction method uses computed values of w and K (Eq 5) which vary slightly with T_x, thereby eliminating part of the influence of a variable V_r. Although the bath-temperature correction method is the most rigorous, it is still sensitive to abrupt changes in thermometer readings.

The effect just described is responsible for the earlier approach of the Federal Blended, Nurse and Pai, and SPC calculations to apparent equilibrium. The final rate, V_r, decreases as the convergence temperature (where $K(B - \theta) = -w$ and $V_r = 0$) is approached. However, if temperature lags cause too large a temperature increase just after introduction of the sample, followed by a drop or an increase which is smaller than it should be, the value of V_r appears to increase instead. This trend causes the correction term V_rT_x to increase with T_x too rapidly, and the corrected rise decreases until equilibrium is attained and V_r falls at the expected rate. Thus, corrected rises calculated during the first 20 min may be too high. In calculation methods which substitute for V_r such time-independent quantities as V_a and w, the decrease in corrected rise during the first 20 min is less protracted because the only agent for this decrease is θ_n, the observed final solution period temperature. It should be remembered, however, that the reason for the abnormal V_r effect is the condition of nonequilibrium. Although the SPC, Federal Blended, and Nurse and Pai methods appear to level off at 10 min, the calorimeter is not really at equilibrium.

For a method to be reproducible from minute to minute, therefore, it is better for the correction term to be independent of the observed final temperature. Methods which use previously determined values for K and rely on w, or V_a, determined from initial rating periods show greater reproducibility within the run. However, their accuracy depends on the correctness of the values for K, w, and V_a. (For example, in the Federal Blended method, an error of 0.001 C in one thermometer reading during the initial rating period will cause an error of 0.006 C in the correction, and an error of 10 per cent in the value of K will cause an error of 0.010 C. In the SPC method, an error in the determination of K or w is equally harmful to accuracy.) Better within-run reproducibility is also obtained if rate calculations are based on



Curve A, calculated by the bath-temperature correction method;
Curve B, same as Curve *A*, but to larger scale; and
Curve C, calculated with an independently measured heat-leakage constant *K*, and stirring energy *w* determined from the initial rating period; bath-temperature corrections made; some scale as Curve *B*.

FIG. 4.—Variation of Corrected Rise with Duration of Solution Period, in a Platinum Precision Calorimeter, Measured with a Platinum Resistance Thermometer.

1.01 ohms = 10 C temperature difference.

temperature readings separated by 10 to 15 min than if they are based on readings 4 or 5 min apart.

Accuracy:

Figure 3 shows the difference between each method and the bath-temperature correction method. Since the latter is the most rigorous, this difference is referred to as "accuracy." The significant portion of each curve lies in the equilibrium period, between 20 and 35 min. If allowance is made for the occasional abrupt variations in the corrected rise, all the methods except the SPC and the Nurse and Pai methods give rises generally within ±0.002 C of the bath-temperature correction method. The latter two methods, however, are consistently high during the equilibrium period.

This systematic difference is due to the integration procedure used in the area correction of the SPC method and the summation in the Nurse and Pai method. The temperatures used in calculating the area *CGHDC* in the SPC method are final temperatures of each interval instead of the average temperatures needed to give the true trapezoidal integral. A similar effect in the Nurse and Pai method causes the correction to be too small. Use of the average temperature for each interval would bring the results of these two methods more closely in line with those of the other methods. It should be emphasized that, in the case of the SPC method, only the calculation procedure is discussed here, not the equipment or calibration techniques.

Comparison of Glass and Metal Calorimeters:

The temperature lags observed in this experiment are characteristic of Dewar flasks because of the large mass of glass which they contain. Time is required for this mass to come to thermal equilibrium with the solution and thermometer. The determination in the Dewar flask may be contrasted with one made with the same ratio of zinc oxide and nitric-hydrofluoric acid mixture in a precision calorimeter constructed almost entirely of platinum 0.01 in. thick, with temperatures measured by a platinum resistance thermometer. The calorimeter is described in reference (12). Curve *A* in Fig. 4 shows the corrected temperature rise for this determination, plotted against the duration of the solution period, as for Fig. 2, calculated by the bath-temperature correction method, and plotted to such a scale that a temperature difference of 0.0001 ohm (about 0.001 C) is readily distinguishable. The curve reaches a constant value at about 8 min (within 0.0005 C from the equilibrium value) and at about 3 min (within 0.002 C, the tolerance used in defining equilibrium for the Beckmann thermometer and the curves in Fig. 2). Thus, equilibrium is attained much earlier in the platinum calorimeter than in the Dewar flask.

Equilibrium comes later, however, if a criterion is chosen consistent with the sensitivity of the platinum resistance thermometer. Curve *B* of Fig. 4 is the same as curve *A*, but plotted to twenty times the scale. Curve *B* does not level off within the limits of T_x for which a corrected rise could be calculated by the bath-temperature correction method, since a 20-min final rating period was used.

However, by using a thermal-leakage constant *K* obtained from several calibration determinations, and a stirring energy *w* obtained from this constant and from the data of the initial rating period, it was possible to make a minute-to-minute calculation of the corrected rise beyond 22 min, plotted as curve *C* in Fig. 4. This curve reached a constant value earlier, at 6 min (within the nearest 0.001 C of the equilibrium value), and at 16 min (within the nearest 0.0001 C). The value of using a separately calculated thermal-leakage constant is evident here again, insofar as minute-to-minute reproducibility is concerned, when the end of the solution period is reached after considerable time due to slow solution of the sample or thermal lag.

SUMMARY AND CONCLUSIONS

Errors involved in measuring the temperature rise in the heat-of-solution methods used for the determination of the heat of hydration of cement may be divided into four groups: (1) errors due to incompleteness of solution, (2) lag errors, due to temperature gradients within the calorimeter, (3) mathematical errors, due to the degree of simplification of the mathematics in the correction calculations for heat leakage and stirring energy, and (4) accidental errors, due to abrupt changes in thermometer readings, of unknown origin or caused by the limited sensitivity of the Beckmann scale.

The methods discussed here are of common mathematical origin, but slight variations in the use of terms and constants or slight departures from mathematical rigor cause differences in the results. In general, all the methods give reasonably accurate results for heat of solution if carried out for a sufficiently long period to prevent lag errors, about 25 min. Within-run reproducibility for various durations of the solution period is best if a calculation is based on constants independent of the duration of the solution period, such as the initial rate V_a or independently determined values of the stirring energy w and the heat-leakage constant K, rather than on such time-dependent quantities as the final rate V_r or a K determined from the final and initial rates of the particular determination. These expedients serve to minimize the effects of all the sources of error listed. Reproducibility is also better for rate measurements taken over long rating periods than over short rating periods, to minimize the effect of abrupt temperature changes.

Reproducibility is no guarantee of accuracy, however, unless the constants are determined accurately. The accuracy of some of the methods, particularly the SPC and Nurse and Pai methods, may be improved by using average interval temperatures instead of initial or final interval temperatures for the integration.

If it is possible to duplicate the errors of a particular method in determining the heats of solution of both dry and hydrated cement, the heat of hydration will be affected only by the corresponding error in the calibration, which will cause only about one-tenth the absolute error in the heat of hydration that it does in the heat of solution.

Although tests have not been made on slowly dissolving substances like pozzolans, the application of the precautions already described would undoubtedly be helpful. If the corrected rise can be calculated from minute to minute by some reproducible method, it should be possible to determine for any experiment a point where subsequent increments of evolved heat may be safely neglected, and to end the solution period at that point.

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CALORIMETRY OF PORTLAND CEMENT
II. APPLICATION OF VARIOUS HEAT-OF-SOLUTION PROCEDURES TO DETERMINATIONS OF HEAT OF HYDRATION

By E. S. Newman[1] and H. A. Berman[1]

Synopsis

The heats of hydration of two cement samples were determined using three types of heat-of-solution calorimeter: the ASTM standard air-bath Dewar, a submerged Dewar with a metal cover, and a submerged metal precision calorimeter using a resistance thermometer. The data were taken in sufficient detail to permit the computation of the corrected temperature rise by eight different methods, all based on Newton's law of cooling, recommended by various authorities in the United States and abroad. The determinations were made in two different rounds of duplicates, and the accuracy and precision of the calorimeters and of the methods are discussed on the basis of the results obtained. Differences among the calorimeters and methods found in heat-of-solution determinations largely disappeared in computing the heats of hydration. By the most rigorous method, standard deviations of 1.45, 1.38, and 0.70 cal per g were found for the heat of hydration as determined by the air-bath Dewar, the submerged Dewar, and the precision calorimeter, respectively.

In Part I is published (**1**)[2] a study of the application of nine calculation procedures as used in various countries to the dissolution of zinc oxide, the standardizing substance, in the cement heat-of-solution calorimeter. This paper is a report of the results obtained by the application of eight of these procedures ((**2–8**) and ASTM Method C 186[3,4]) to the replicate determinations of the heats of hydration of two cements distributed by the ASTM Working Committee on the heat of hydration.

In heat-of-solution determinations, there are three major sources of error in measuring the temperature rise, namely (1) calculation errors due to the application of various simplified approximate methods to the rigorous computation of the corrected temperature rise, (2) possible errors due to slow or incomplete solution of some cements, and (3) thermal-lag errors due to the slow establishment of equilibrium temperature gradients between the interior of the calorimeter and the surroundings, or due to the presence of heat sinks in the calorimeter. These lag errors are particularly large in the glass Dewar vessels almost universally used in cement calorimetry. No attempt is made to evaluate separately these kinds of errors in this paper, the chief purpose of which is to permit an over-all comparison of the results obtained by the application of all the various standardized calculation procedures to a single group of experimental determinations.

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[1] National Bureau of Standards, Washington, D.C.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

[3] Method of Test for Heat of Hydration of Portland Cement (C 186 – 55), 1961 Book of ASTM Standards, Part 4.

[4] Method of Test for Heat of Hydration of Portland Cement (C 186 – 49), 1952 Book of ASTM Standards, Part 3.

MATERIALS, APPARATUS, AND PROCEDURE

Two samples of cement, one of Type II portland and one of portland blast-furnace slag cement, had been distributed to a number of laboratories for cooperative tests of the heat of hydration. Duplicate heat-of-solution determinations were made at the National Bureau of Standards with the unhydrated cement and with the hydrated pastes at 7 and 28 days, using the Beckmann thermometer and the air-bath Dewar described in the Federal (2) and ASTM[3] specifications, a Beckmann thermometer with a submerged Dewar flask with a single metal cover, and a precision submerged metal calorimeter (9) with a platinum resistance thermometer. The entire group of tests was repeated in a second round a few days later, making 72 heat-of-solution tests in all. The two submerged calorimeters were operated in a water bath controlled to ±0.002 C during a test. Temperature readings of the two Dewar calorimeters were taken during the tests on a schedule meeting the combined requirements of the three methods of calculation given in the Federal Specification, that is, at times of -10, -5, 0, 1, 2, 3, 4, 5, 10, 15 min, and so on, where zero time represents the time that introduction of the sample is begun. Temperature readings of the precision calorimeter were taken every 2 min except during the time 0 to 10 min, when they were taken every minute. All determinations were extended to at least 40 min.

TABLE 1.—CHARACTERISTICS OF VARIOUS HEAT-OF-SOLUTION PROCEDURES FOR CEMENTS.

Method	Duration of Rating Periods, min		Duration of Solution Period, min	Reading Interval, min	Reference
	Initial	Final			
1. ASTM Method C 186 – 55 . . .	0	20	20	20	footnote 3
Federal Regular	0	20	20	20	(2)
2. Federal Alternate	5	15	$25^{a,b}$	5^{c}	(2)
ASTM Method C 186 – 49 . . .	5	15	$25^{a,b}$	5^{c}	footnote 4
3. Federal Blended	10	0	30	5^{c}	(2)
4. BS 1370:1958	5	7 to 8	12 to 17^{d}	1	(3)
5. BRS C548	10	0	20	20^{e}	(4)
6. Nurse and Pai	0	0	10	1	(5)
7. CERILH	4	4	4 to 10^{f}	1	(6)
8. SPC	0	0	^{g}	5^{c}	(7)

[a] Minimum arbitrarily set at 25 min in this laboratory. Maximum 45 min by specification.

[b] Extended until rate per 5-min constant to ±0.001 C for 15 min.

[c] One-minute intervals during first 5 min of solution period.

[d] In examples given.

[e] Readings taken only at -10, 0, and 20 min.

[f] As determined by attainment of constant rate to ± 0.003 C per min.

[g] As determined by conformity of rate of temperature rise to normal rate without dissolution of sample as previously determined in a separate experiment.

Hydrated cement samples were prepared of sufficient size that all determinations in a single round with a given cement at a given age were made with material taken from a single vial of hardened paste. Thus comparisons among calorimeters or methods of calculation do not include variability introduced by differences in mixing, aging, or grinding the paste. For each heat-of-solution determination a sample was taken for determination of loss on ignition. These samples of hardened paste were dried for a minimum of 1 hr at 105 C and then ignited overnight at 950 C. Heats of solution were calculated on an ignited basis, as described in the Federal and ASTM specifications.

TABLE 2.—EQUATIONS USED IN CALCULATING CORRECTED TEMPERATURE RISE OF HEAT-OF-SOLUTION CALORIMETER.

Method	Equation[a]
1. ASTM Method C 186 – 55 Federal Regular	$R_c = (\theta_{20} - \theta_0) - (\theta_{40} - \theta_{20})$
2. Federal Alternate ASTM Method C 186 – 49	$R_c = (\theta_n - \theta_0) - V_r T_x - k(\theta_r T_x - \theta_x T_x)$
3. Federal Blended[b]	$R_c = (\theta_{30} - \theta_0) - V_a T_x - K(\theta_a T_x - \theta_x T_x)$
4. BS 1370:1958	$R_c = (\theta_n - \theta_0) - \sum_1^n c_i^*$
5. BRS C548	$R_c = (\theta_{20} - \theta_0) + 2K^*(\theta_{20} - \theta_0) - 2(\theta_0 - \theta_{-10})$
6. Nurse and Pai	$R_c = (\theta_{10} - \theta_0) - \sum_1^{10} C_i^*$
7. CERILH	$R_c = (\theta_n - \theta_0) - \sum_1^n c_i$
8. SPC	$R_c = (\theta_n - \theta_0) - WT_x - K(B_0 T_x - \theta_x^* T_x)$

[a] For meaning of symbols, see Appendix, p. 860.

[b] V_a and θ_a are averaged over a 10-min initial rating period in the Federal Blended method.

HEAT-OF-SOLUTION COMPUTATIONS

In Table 1 are listed the characteristics of eight of the various procedures discussed at length in the previous paper (1). In Table 2 are listed the equations by which the corrected temperature rises are calculated by the various procedures, the symbols and their explanations being listed in the Appendix.[5] Because of the way in which the data were taken, interpolations were necessary in making some of the calculations. Several of the methods require 1-min readings of the calorimeter temperature, but these were available only during the first 5 min of the solution period (10 min for the precision calorimeter). Linear interpolation was used, and fortunately during the times when it was required the calorimeter temperature was changing either uniformly or slowly so that interpolation errors were small.

Experience has shown that the bath-temperature correction method discussed in reference (1) gives results identical to those obtained with the Federal Alternate method when the bath temperature is constant to ± 0.005 C, even when the precision calorimeter is used. Consequently, computations according to the bath-temperature correction method are not included in the tables.

The heats of solution calculated by the eight methods were subjected to systematic statistical analysis to find the effects of method of calculation, of round of test, and of calorimeter. Heats of hydration were determined by subtracting individual heats of solution of hydrated paste from the corresponding heat of solution of an unhydrated cement sample, in the order in which the determination was made, rather than by subtracting averages. These data also were analyzed statistically.

RESULTS AND DISCUSSION

Calibrations:

The heat capacities to be used in

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[5] See p. 860.

computing the heats of solution of the cements were themselves calculated by the individual methods to be used for the cements. Zinc oxide was the standardizing substance for all three calorimeters, using the value 256.1 cal per g at 30 C (2), although the precision calorimeter could have been calibrated electrically. The Dewar calorimeters were calibrated three times each, and the precision calorimeter five times. Average values and standard errors are listed in Table 3.

TABLE 3.—HEAT CAPACITIES OF CALORIMETERS, CALCULATED BY VARIOUS PROCEDURES.

Calorimeter, Method of Calculation	Air-Bath Dewar		Submerged Dewar		Precision	
	Heat Capacity, cal per deg C	Standard Error,[a] cal per deg C	Heat Capacity, cal per deg C	Standard Error, cal per deg C	Heat Capacity,[b] cal per ohm	Standard Error,[a] cal per ohm
1. Federal Regular ASTM Method C 186 – 55	397.45	0.33	393.22	0.08	6647.3	1.47
2. Federal Alternate ASTM Method C 186 – 49	397.87	0.11	393.85	0.11	6664.3	1.36
3. Federal Blended	397.95	0.38	393.92	0.06	6663.6	1.86
4. BS 1370	397.49	0.15	393.37	0.09	6648.8	1.66
5. BRS C548	397.05	0.11	393.20	0.06	6647.3	1.52
6. Nurse and Pai	397.37	0.20	393.16	0.25	6649.5	1.37
7. CERILH	397.78	0.42	393.25	0.08	6662.5	1.20
8. SPC	397.82	0.18	393.65	0.15	6650.3	1.51

[a] Standard error = $\sqrt{(\Sigma x^2 - (\Sigma x)^2/n)/n\,(n - 1)}$

[b] $\Delta R/\Delta\theta$ = 0.1010 ohm per deg C at 25 C.

TABLE 4.—HEATS OF SOLUTION CALCULATED BY VARIOUS PROCEDURES.[a]

Cement	Method	Air-Bath Dewar			Submerged Dewar			Precision Calorimeter		
		Dry	7-Day	28-Day	Dry	7-Day	28-Day	Dry	7-Day	28-Day
Portland	1	608.47	538.95	528.08	609.36	539.60	527.16	610.43	541.38	529.53
	2	608.00	538.96	527.23	608.55	540.05	527.21	609.44	540.74	529.17
	3	608.58	540.01	528.13	610.91	543.44	532.03	608.71	540.74	528.54
	4	608.42	538.68	529.03	608.51	538.90	526.93	609.16	540.95	529.24
	5	608.18	539.23	527.97	610.85	542.04	530.20	610.14	540.80	528.87
	6	607.62	539.06	527.64	608.64	540.30	528.49	608.98	540.85	529.12
	7	607.42	537.28	526.72	607.80	538.76	525.10	607.07	540.12	527.93
	8	609.00	539.31	528.80	607.43	538.03	525.07	609.22	540.70	528.33
Portland blast-furnace slag	1	583.30	519.74	504.24	583.01	519.38	503.76	585.40	521.58	506.58
	2	582.94	519.87	504.24	582.83	519.33	503.80	584.97	521.26	506.16
	3	583.41	521.50	505.60	583.00	523.18	509.18	585.31	521.64	506.22
	4	582.98	519.59	504.34	582.58	519.65	503.57	584.71	521.18	505.88
	5	583.69	521.22	505.30	583.08	521.94	507.02	585.73	521.68	506.27
	6	582.80	519.91	504.38	582.62	520.12	504.90	585.02	521.15	505.98
	7	582.37	518.08	502.57	581.94	517.77	502.49	582.97	519.46	504.08
	8	584.99	520.88	504.83	581.88	517.36	502.17	584.61	521.42	507.00

[a] Averages of four determinations, cal per g ignited weight.

Examining first the standard errors, it appears that when only one calorimeter is considered at a time all methods are of about equal precision when applied to calibrations. Expressed as coefficient of variation, values of about 0.06, 0.03, and 0.02 per cent were found for the air-bath Dewar, the submerged Dewar, and the precision calorimeter, respectively.

TABLE 5.—STANDARD DEVIATION OF HEATS OF SOLUTION CALCULATED BY VARIOUS PROCEDURES.[a]

Cement	Method	Air-Bath Dewar			Submerged Dewar			Precision Calorimeter		
		Dry	7-Day	28-Day	Dry	7-Day	28-Day	Dry	7-Day	28-Day
Portland	1	1.28	0.87	1.23	1.04	0.24	0.94	0.41	0.39	0.26
	2	1.02	0.65	1.71	1.15	1.04	0.84	0.45	0.66	0.34
	3	0.74	0.78	0.37	3.42	0.96	1.33	0.57	0.59	0.36
	4	0.78	1.48	0.90	1.16	2.15	0.98	0.66	0.38	0.32
	5	0.59	0.43	0.95	2.53	0.57	0.63	0.28	0.33	0.73
	6	0.46	0.29	1.11	1.94	0.44	0.34	0.67	0.35	0.36
	7	0.77	1.79	1.41	0.91	1.02	1.42	0.87	0.38	0.23
	8	0.97	0.96	1.96	0.98	1.27	1.20	0.36	0.42	1.38
Portland blast-furnace slag	1	0.65	0.68	0.88	0.50	0.59	0.53	0.30	0.63	0.29
	2	0.99	0.92	1.03	0.43	0.93	0.38	0.23	0.52	0.29
	3	1.75	0.93	1.17	0.56	2.54	0.78	1.04	0.95	0.36
	4	1.16	0.88	0.89	0.47	0.94	0.53	0.16	0.52	0.31
	5	1.30	0.70	0.95	0.51	1.49	0.62	0.72	0.72	0.34
	6	0.63	0.71	0.60	0.40	1.09	0.52	0.36	0.66	0.22
	7	1.15	0.69	0.99	0.49	1.23	1.37	0.51	0.51	0.33
	8	1.12	1.19	1.38	0.49	1.43	0.14	1.28	0.54	0.45

[a] Standard deviation = $\sqrt{(\Sigma x^2 - (\Sigma x)^2/n)/(n - 1)}$; cal per g ignited weight.

TABLE 6.—HEATS OF HYDRATION CALCULATED BY VARIOUS PROCEDURES.[a]

Cement	Method	Air-Bath Dewar		Submerged Dewar		Precision Calorimeter	
		7-Day	28-Day	7-Day	28-Day	7-Day	28-Day
Portland	1	69.52	80.39	69.76	82.20	69.05	80.90
	2	69.04	80.77	68.50	81.34	68.70	80.27
	3	68.57	80.45	67.47	78.88	67.97	80.17
	4	69.74	79.39	69.61	81.58	68.21	79.92
	5	68.95	80.21	68.81	80.65	69.34	81.27
	6	68.56	79.98	68.34	80.15	68.13	79.86
	7	70.04	80.70	69.04	82.70	66.95	79.14
	8	69.69	80.20	69.40	82.36	68.52	80.89
Portland blast-furnace slag	1	63.56	79.06	63.63	79.85	63.82	78.82
	2	63.07	78.70	63.50	79.03	63.71	78.81
	3	61.91	77.81	59.82	73.82	63.67	79.09
	4	63.41	78.64	62.93	79.01	63.53	78.83
	5	62.47	78.39	61.14	76.06	64.05	79.46
	6	62.89	78.42	62.50	77.72	63.87	79.04
	7	64.29	79.80	64.17	79.45	63.51	78.89
	8	64.11	80.16	64.52	79.71	63.19	77.61

[a] Calories per gram ignited weight.

Statistical examination of the heat capacities in Table 3 shows that only the BRS C548 method gave average values significantly lower than the Federal Alternate, which may be considered a standard here since it contains the fewest short-cuts and simplifying assumptions. The Federal Regular method (ASTM C 186 – 55) consistently gave low values, due to the systematic omission of the $K(\theta_r T_x - \theta_x T_x)$ term. Deviations of the observed sizes however, even when significant or systematic, are of minor importance because whatever the value of the heat capacity obtained, it is used in computing the heat of solution of both the hydrated and unhydrated cement. An error of 0.2 per cent in heat capacity will therefore cause a systematic error of only 0.2 per cent in the heat of hydration, about one-tenth the observed standard deviation of the latter.

TABLE 7.—STANDARD DEVIATIONS OF HEATS OF HYDRATION CALCULATED BY VARIOUS PROCEDURES.[a]

Cement	Method	Air-Bath Dewar		Submerged Dewar		Precision Calorimeter	
		7-Day	28-Day	7-Day	28-Day	7-Day	28-Day
Portland	1	0.60	1.33	1.09	1.64	0.59	0.24
	2	0.55	1.54	1.66	1.73	0.80	0.29
	3	0.62	1.14	4.18	4.19	0.95	0.58
	4	2.07	1.27	1.54	1.59	0.50	0.53
	5	1.07	1.25	2.70	2.91	0.18	0.31
	6	0.72	1.50	2.10	2.46	0.53	0.66
	7	1.84	1.54	0.33	1.92	0.56	0.77
	8	0.66	2.58	1.76	1.57	0.65	1.31
Portland blast-furnace slag	1	0.50	1.34	0.82	1.02	0.60	0.38
	2	1.52	1.85	1.10	0.81	0.43	0.29
	3	1.72	0.59	1.84	1.12	0.58	0.77
	4	1.58	1.27	1.23	0.99	0.64	0.46
	5	1.25	0.54	1.36	1.08	0.36	0.43
	6	0.82	0.71	1.14	0.90	0.72	0.33
	7	1.71	2.09	1.11	1.73	0.75	0.68
	8	2.25	1.64	1.93	0.45	1.77	1.39

[a] Standard deviation = $\sqrt{(\Sigma x^2 - (\Sigma x)^2/n)/(n - 1)}$; cal per g ignited weight.

Heats of Solution:

Table 4 presents a summary of the heats of solution of the cement samples. Each entry in this table is the average of the calculated values for four determinations of heat of solution, two made on one day on one paste (in the case of hydrated cement) and two made on a second day with a different paste. Variance analysis showed no significant difference between the two pastes, or between determinations on unhydrated cement made on different days. The four individual values were used in calculating the standard deviations given in Table 5.

The results in Table 4 were averaged, and the standard deviations in Table 5 were pooled to obtain single figures representing the methods and the calorimeters. The Federal Blended, BRS C548, SPC, and CERILH methods were all somewhat less precise than the others, and the first two of these gave consistently larger heats of solution. On the other hand, the CERILH method gave consistently low values for the heats of solution. The remaining four methods of calculation, the Federal Alternate, ASTM C 186 – 55 (Federal Regular), BS 1370, and the Nurse and Pai methods, all gave essentially both the same average heat of solution and the same precision (standard deviation about 0.5 cal per g). On the basis of these heat-of-solution tests, then, there seems to be little to choose among these four methods. This conclusion is an extension of that of Parker and Nurse (8) who found little difference between the results of British and American tests.

The precision calorimeter gave heats of solution about 1 cal per g higher than the values obtained with the other two calorimeters, which are approximately equal.

Heats of Hydration:

The heats of hydration obtained from the average values in Table 4 are given in Table 6. Table 7 gives the standard deviations of the heats of hydration calculated by pairing, in the order of determination, the individual heats of solution of the dry cements with the individual determinations made with the corresponding hydrated pastes.

The heats of hydration in Table 6 were averaged, and the standard deviations in Table 7 were pooled in order to obtain single figures representing the methods of calculation and the calorimeters. In determining the values of the heat of hydration, the compensation of corresponding errors in the heats of solution of hydrated and unhydrated cement has an opportunity to operate. For this reason the heats of hydration determined by seven of the eight methods and on all three calorimeters are essentially equal. The Federal Blended method gave low results and was distinctly more variable than the others. There is some evidence that the BRS C548 and the Nurse and Pai methods give somewhat lower results, and the ASTM C 186 – 55 (Federal Regular), CERILH, and SPC methods somewhat higher, than the Federal Alternate and BS 1370 methods, but the differences are insignificant. Standard deviations of 1.45, 1.38, and 0.70 were obtained by method 2 for the heat of hydration as determined by the air-bath Dewar, the submerged Dewar, and the precision calorimeter, respectively.

Sources of Variability:

Four of the methods, the Federal Blended, BRS C548, Nurse and Pai, and the SPC, were designed to be used with pozzolanic or slag-containing cements containing slowly dissolving or insoluble material. The first three of these methods base the determination on a calibration value of K and an initial rating period, dispensing with a final rating period, and the SPC method depends completely on the constants obtained during calibration. The first three are sensitive, therefore, to errors in determining the initial rate. Only the Federal Blended method suffered from this fault in these tests. Examination of the original data showed that most of the variability in the heat of hydration measured by this method arose from the fact that the initial rates of two of the eight unhydrated cement determinations made in the submerged Dewar were abnormally small. Since the same determinations were the basis of the calculations by all methods, the effect of these two measurements was also apparent in the results for the Nurse and Pai method.

One of the cements tested in this program was a portland blast-furnace slag cement. However, in dissolution this cement did not show the slow and long-continued temperature rise frequently characteristic of these cements. For this reason, all results were combined. If a more typically behaving slag cement had been used, there would probably have been substantial differences between the results obtained with it by the four methods intended for slowly dissolving cements and the four suitable only for more rapidly dissolving material.

Length of the Solution Period:

There are a number of tests that can be used to indicate the end of the solution period. The most accurate of these, the constancy of the corrected rise after dissolution of the sample is complete, is discussed at length in the previous paper (1). The necessary computations are long and involved. A second test, the simplest, is the attainment of a constant rate of temperature rise at the end of the solution period. The authors believe that this test is open to misinterpretation, and have discussed it elsewhere (1). A third, relatively simple test based on Newton's law of cooling is described as follows.

This test for completion of the solution period, particularly useful with Beckmann thermometers, is the calculation of the final rate to be expected by the approximate equation $K^* = (V_a - V_r)/(\theta_n - \theta_0)$ rearranged as $V_r = V_a - K^*(\theta_n - \theta_0)$. The value of K^* can be calculated from calibration experiments, and the other quantities are easily found from the data for the experiment being tested. The determination should be continued until an appropriate value of V_r is obtained, whereupon the values of k, w, and the corrected rise, R_c, can be calculated.

With pozzolanic or slag cements dissolution is frequently incomplete in a time suitable for calorimetry, and the above tests cannot be applied. Recourse must then be taken to the approximate methods, omitting or calculating a final rating period.

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(See Appendix on page 860.)

APPENDIX

SYMBOLS USED IN CALCULATIONS OF CORRECTED TEMPERATURE RISES

R_c, Corrected temperature rise in deg C

$\theta_0, \theta_5, \cdots, \theta$ is the calorimeter temperature in deg C. Numerical subscripts indicate the time in minutes relative to the beginning of the sample introduction. Subscript n indicates the nth minute at the end of the solution period when its length is not fixed by the specification but by the behavior of the calorimeter. Subscripts a and r indicate the average calorimeter temperature during the initial, a, or final, r, rating period

V_a, V_r, The average rate of temperature rise in deg C per min or in deg C per 5 min during the initial or final rating periods.

T_x, The duration of the solution period, either in minutes or in 5-min periods, depending on the method

k, The heat-leakage constant calculated from the data of the individual determination being computed, $(V_a - V_r)/(\theta_r - \theta_a)$. Units are min^{-1}, or 5 min^{-1}, if V_a and V_r are in deg C per min or deg C per 5 min respectively

K, The heat-leakage constant calculated from one or more calibration experiments

k^*, The individual heat-leakage constant calculated by the equation $k^* = (V_r - V_a)/(\theta_n - \theta_0)$ (British methods)

K^*, The average heat-leakage constant calculated from one or more calibration experiments (British methods)

θ_x, θ_x^*, The average calorimeter temperature during the solution period. Appears only in the product $\theta_x T_x$, the area under the time-temperature curve in C deg-min or in C deg- (5-min periods), depending on the method. The asterisk refers to the SPC method in which $\theta_x^* T_x$ differs from the trapezoidal area by the quantity $\theta_x^* T_x - \theta_x T_x = (\theta_n)/2 - 0.1(\theta_0 + 4\theta_5)$

T_a, T_r, Duration of the rating period in minutes or in 5-min periods, depending on the method

c, c^*, The correction for a 1-min period determined from a plot of V versus θ (or $(B - \theta)$) from the data of an individual determination. An asterisk indicates that the temperature at the end of the 1-min period is used. The absence of an asterisk indicates the use of the average temperature during the period

C, C^*, The correction for a 1-min period determined from a plot of V versus θ (or $(B - \theta)$) from calibration experiments with no material or with rapidly dissolving material

W, The stirring correction in deg C per min or deg C per 5 min calculated from one or more calibration experiments. $W = V - K(B - \theta)$

w, The stirring correction calcuated from the data of an individual determination.

B_0, B_a, The bath temperature or the room temperature when no bath is used. Subscripts have the same meaning as for θ, T, and so on.

SIGNIFICANCE OF TESTS FOR HEAT OF HYDRATION OF CEMENT*

By W. J. McCoy[1]

When portland cement is mixed with water a reaction which results in the setting and hardening of the paste takes place; this reaction is called hydration. When this reaction occurs, heat is generated as it is an exothermic reaction; the rate and amount of heat liberation are functions of the composition and fineness of the cement. In most concrete construction this heat is dissipated and is of little concern; however, in mass concrete such as dams, large abutments, and massive foundations, if proper precautions are not taken, a considerable rise in temperature of the concrete accompanied by expansion can occur because of the relatively low thermal conductivity of the concrete. If the temperature of the concrete increases excessively it can result in cracking at a later time when the mass cools and contracts. This heat evolution characteristic of hydrating cement is not necessarily detrimental—it can be used to advantage during cold weather. Studies prior to the construction of the Hoover Dam gave special emphasis to the effect of cement composition and fineness on the quantity of heat evolved during hydration (**1**).[2]

The principal compounds of portland cement hydrate at different rates. Generally speaking and under ordinary conditions the tricalcium aluminate (C_3A) releases most of its heat in the first day or so, the tricalcium silicate (C_3S) in the first few weeks; the dicalcium silicate (C_2S) and calcium ferroaluminate (C_4AF) hydrate more slowly. Studies reported by Lerch and Bogue (**2**) show that the compounds associated with the composition of portland cement not only hydrate at different rates but also have considerably different heats of hydration in terms of calories per gram of hydrated compound as shown in Table 1.

These are heat of hydration values for the pure compounds when completely hydrated. It should be noted that compounds such as C_3S and C_2S do not occur in portland-cement clinker in the pure form but occur in the form of alite and belite which are essentially C_3S and C_2S associated with small amounts of other compounds. For this and other reasons the heat of hydration of the calculated potential compounds in portland-cement clinker differs somewhat from the values noted in the foregoing table but the relative order of magnitude is believed to be the same.

TABLE 1.—HEATS OF COMPLETE HYDRATION OF CEMENT COMPOUNDS.

Compound	Heat of Hydration, cal per g
C_3S	120
C_2S	62
C_3A	207
C_4AF	100
Free CaO	279
Free MgO	203

As previously mentioned, fineness of cement is also an important factor since

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* Presented at the Sixty-sixth Annual Meeting of the Society.

[1] Director of Research, Lehigh Portland Cement Co., Allentown, Pa.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

TABLE 2.—AVERAGE HEATS OF HYDRATION OF DIFFERENT TYPES OF PORTLAND CEMENT.

Cement	Heat of Hydration, cal per g; 70 F Storage					
	3 days	7 days	28 days	3 months	1 yr	6½ yr
Type I	61	80	96	104	109	117
Type II	47	61	80	88	95	98
Type III	75	92	101	107	114	121
Type IV	41	51	66	74	81	85
Type V	44	55	74	83	91	94

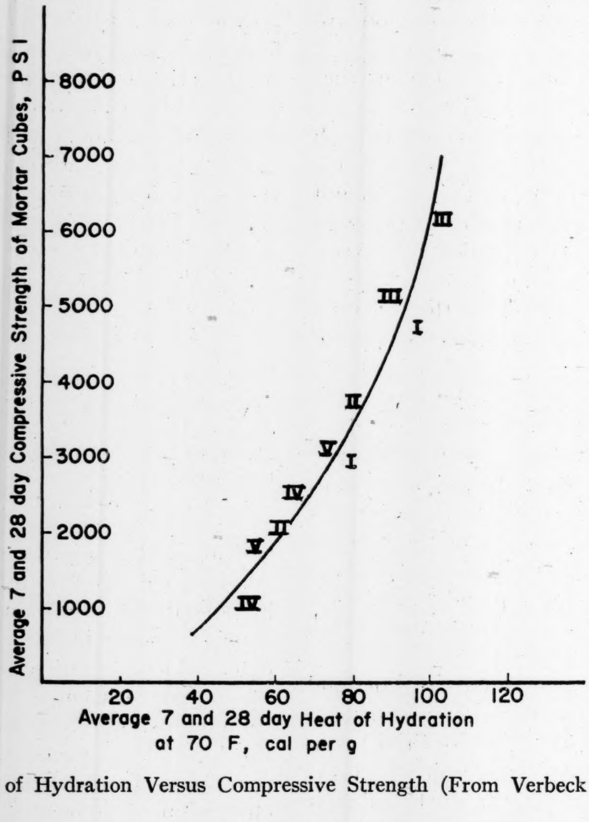

Fig. 1.—Heat of Hydration Versus Compressive Strength (From Verbeck and Foster (3 p. 1253)).

Numbers along curve refer to the type of cement.

it has a significant effect on the rate of hydration at early ages and it will have a similar effect on the magnitude of the heat of hydration.

Table 2 gives average heat of hydration values for the various types of portland cement as reported by Verbeck and Foster (3).

If consideration is given to the relative amount of the potential compounds characteristic of the various type cements, the above heat of hydration values for the different type cements are in the same order of relative magnitude that would be predicted based on the heat of hydration values for the compounds noted in Table 1. The effect of fineness accentuates the difference between the values for type III and each of the other types. It is interesting to note that in all cases, including type III, the cements are continuing to hydrate even at the age of $6\frac{1}{2}$ yr. On this basis one would expect cements to continue to gain in strength comparable to this gain in heat of hydration. Verbeck and Foster (3, p. 1253) published the curve in Fig. 1 to show there is a good correlation between heat of hydration and strength for each of the five types of cement both at 7 days and at 28 days.

The relative positions of the various type cements on the strength - heat of hydration curve are as would be expected with type III at the top, then type I, then II, with V and IV last in that order. These are the average values for the cements in the Portland Cement Assn. long-time test series.

Methods for Determining Heat of Hydration

The three principal methods for determining the heat of hydration are (1) use of an adiabatic calorimeter, (2) use of a conduction calorimeter, and (3) the heat of solution method. Only brief mention will be made of the first two methods since they are used primarily for research purposes. The adiabatic calorimeter maintains the temperature of the container the same as that of the hydrating cement, and the temperatures are recorded against time. The conduction calorimeter usually consists of four principal parts, namely, a copper cup to contain the cement paste, a brass tube to conduct the heat away from the specimen cup, resistance thermometers attached to the two ends of the conduction tube, and a heat-dispersing base immersed in a water bath. The specimen cup is placed on a copper pedestal and the apparatus is placed in a thermos to minimize heat loss by radiation and air convection. In this procedure practically all the heat generated by the hydration of the cement in the specimen cup is conducted to the water bath. The difference in temperature between the specimen cup and the water bath is measured. This indicates the rate at which heat is conducted away from the specimen or the rate of hydration.

Heat of Solution Method:

The heat of solution method for determining heat of hydration has proved to be the most practical from a specification standpoint. The essentials of this method are to dissolve a small sample of the unhydrated cement in nitric-hydrofluoric acid solution in a thermos container and determine the heat of solution of the unhydrated cement in terms of calories per gram. Then neat mixes of the cement are hydrated for a given period of time such as 7 or 28 days and the hydrated sample is pulverized and its heat of solution determined. After correcting for a number of factors, the heat of hydration of the cement for the time involved is calculated by subtracting the heat of solution value for the hydrated cement from the similar value for the unhydrated cement. One of the first papers containing a description of this type of procedure was in 1932 by Woods, Steinour, and Starke (4). This method was further developed and explained by Lerch in 1934 (5).

A heat of solution method for determining heat of hydration of portland cement was adopted as an ASTM tentative in 1944 and adopted as standard in 1947. Soon thereafter the Subcommittee on Heat of Hydration of ASTM Committee C-1 on Cement, in cooperation with Edwin S. Newman of the National Bureau of Standards, investigated a new simplified heat of solution method. Since the preliminary tests indicated the simplified method to be promising, a cooperative test program among several laboratories was conducted in 1951. This program included a number of cement samples that were tested for heat of hydration by both the proposed rapid method and the ASTM standard method which at that time was C 186 – 49.

The results of this series showed the rapid method to have the same precision as the present standard method. The average deviation among the laboratories was of the order of 2 to 3 cal per g and within a given laboratory was of the order of 1 to 2 cal per g. The essential difference between the two methods is this: In the short method only three readings are taken on the Beckmann thermometer—one when the sample is introduced, one at 20 min (end of the solution period), and one again at 40 min, which is called the rating period and is used as the basis for the temperature correction. In the old standard method the readings were taken every minute after introduction of the sample for a while and then every 5 min until a constant temperature was attained. The calculations involved to obtain the temperature rise with these many readings were complex and very lengthy. At the conclusion of these studies the rapid method was published as tentative, and after a trial period it was adopted as standard in 1955[3] and the old standard method removed from the books. A similar change was made in the Federal Specifications so that the method for determining heat of hydration is now the same for both Federal and ASTM specifications.

Papers by Newman and Wells (6) and by Nurse (7) indicated that it would be feasible to determine the heat of hydration of portland blast-furnace slag cement by the present ASTM Method C 186. The Subcommittee on Heat of Hydration in 1957–1958 sponsored cooperative test programs and studies to provide first-hand information as to whether the heat of hydration of the portland blast-furnace slag cements covered by ASTM Specification C 205[4] could be satisfactorily determined by the present method for portland cement. It was concluded that Method C 186 works as well with portland blast-furnace slag cement as with portland cement.

These studies confirmed previous ones which showed a need to improve the reproducibility of the test method and to reduce the deviation of results among laboratories. The present activities of the Subcommittee on Heat of Hydration are focused on this problem. The most recent modification being investigated is the use of a constant-temperature water bath for immersion of the thermos jar so as to obtain better temperature control than is provided by the present Method C 186. However, the results to date have not been promising since there appears to be no significant improvement in the reproducibility of the method effected by the use of a constant-temperature water bath.

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[3] Method of Test for Heat of Hydration of Portland Cement (C 186), 1961 Book of ASTM Standards, Part 4.

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SIGNIFICANCE OF TESTS FOR FINENESS*

By Bruce E. Kester[1]

Synopsis

Testing of fineness of particulate materials ranges from early simple screening procedures through a wide variety of procedures based on visual observation enhanced by microscopic techniques, derivations of Stokes' law, permeability, adsorption, and the newest method of electronic size analysis. Limitations and advantages of each of these systems are considered.

The influence of fineness on the properties of cement are examined in some detail with comparison of resultant fineness gradations from open and closed circuit grind. The principal influence is noted as rate of strength development rather than ultimate strength. Paralleling the rate of strength development is the rate and maximum heats of hydration as influenced by fineness. Workability and bleeding also are governed by fineness although it is pointed out that the many other variables of cement manufacture may be of equal or even greater importance in determining performance characteristics.

Fineness is of major significance on performance properties of portland cement and fineness testing is most properly a manufacturing control tool seeking uniformity of product and constant performance characteristics.

Two statements represent the historical significance of fineness: "Fine grinding materially improves the quality of the cement itself" and "it is the softer pieces, or those which have received the least calcination that are ground finest." These statements by Faija (**1**)[2] published in 1881 still reflect the common opinion and to some extent the practice of modern cement industry.

However, the degree of fineness referred to has undergone far greater transformation than the characteristics ascribed to high fineness. The specification of 1881 required 100 per cent passing a 25-mesh sieve and 85 per cent passing a 50-mesh sieve with a 400-psi tensile strength requirement at 7 days and 500 psi at 28 days, certainly respectable values when compared to ASTM Specifications C 150[3] where fineness must now be measured in specific surface expressed as square centimeters per gram as 2800 by the air permeability test and the optional tensile strength requirements of 275 psi at 7 days and 350 psi at 28 days.

Since strength is usually considered synonymous with quality, either the statement "fine grinding materially improves quality" is untrue or modern cements must be suspect as regards their degree of calcination. Fortunately neither premise is completely true and refinements in techniques and knowledge have advanced the art of cement making to a point where the influence of fineness is more precisely known and burning techniques with proper mix control have virtually eliminated unsound undercalcined cements.

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* Presented at the Sixty-sixth Annual Meeting of the Society.

[1] Technical Director, Missouri Portland Cement Co., St. Louis, Mo.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

[3] Specifications for Portland Cement (C 150), 1961 Book of ASTM Standards, Part 4.

The most readily apparent influence of fineness on modern cement clinker grinding is in the rate of strength development. While this effect is not noted in the early reference quoted, nor could such an opinion be gleaned from a detailed perusal of Specifications C 150, type III, the high-early-strength cements, are universally ground to nearly twice the specific surface of type I, the normal strength development cement. Heat of hydration will also parallel the rate of strength development.

Like the traditional organic chemist's reliance on the premise that every 10 C rise in temperature doubles the reaction rate, the cement-maker relies on doubling the reaction rate (hydration of cement) by doubling the available surface for reaction.

Workability—still an intangible, noted and frequently complained of by the craftsman working in concrete—bears a relationship to fineness as does the water requirement for equivalent plastic deformation properties of the cement paste.

The bleeding of cement pastes in mortar and concrete shows a correlation to fineness although this property may be altered by the presence or lack of entrained air, by aggregate gradation, by the use of water-reducing admixtures, and even by climatic conditions.

Later sections of this paper will deal in greater detail with these properties, but consideration must be given to the question of whether fineness is a controlling factor or is controlled by other cement properties. Some review of the tools of fineness measurement is required to establish the degree of reliability of either control of the manufacturing process or of valid specifications.

Since in the author's opinion fineness governs performance properties of cement, it in itself is not a suitable specification item. The performance characteristics themselves—strength, rate of strength development, shrinkage, expansion, bleeding, and so on—should be made the governing specification items. Every cement manufacturer who has exchanged views of fineness with the author uses fineness as a manufacturing control tool. Current fineness limits of type I cement, for example, do not reflect the 2600 minimum to 2800 average specific surface by air permeability. In examination of 109 type I cement samples collected over a twelve-state area, these cements averaged 3350 specific surface, with a low value of 3000 and a high of over 4200. The fineness levels, considerably above specification requirements, are imposed by 3-, 7-, and even 28-day strength requirements. Again these strength specifications are not realistically related to ASTM Specification C 150 as the 3-day strengths were 115 per cent in excess of specification, 7-day strengths 98 per cent in excess, and 28-day strengths 65 per cent in excess in these same 109 samples.

Uniformity of product has become of increasing importance, with more concrete construction under closer inspection; with larger concrete contracting concerns using different brands of cements from different or adjacent market areas; and with the general competitive cement market situation. Fineness control is one of the easiest of all control systems in the cement manufacturing process. By close attention to mill efficiency and frequent sampling it is possible, through mill feed rate control, to control product fineness manually to close tolerances. With the advent of recent automatic mill load sensing devices based on mill noise levels, mill output rates sensed by nuclear measuring devices, and power consumption levels of elevators, separators, or other auxiliaries, control systems produce remarkably uniform fineness results by keeping a relatively constant grinding load.

If quality control systems can effectively blend and control raw materials and can uniformly calcine, sinter, and quench the kiln feed, then fineness control assures uniformity of product. If potential compound composition is not held under rigorous control during raw material proportioning and blending; if the reactions sought during burning are not uniformly controlled and completed; if the quenching of the kiln discharge clinker is not rapid and inclusive of all clinker produced, then fine grinding by itself cannot and will not enhance the quality of cement so produced.

THE TESTING OF FINENESS

While fineness was not measured by the Romans in their pozzolanic cements or even by Aspdin, it was known that to create a hydraulic cement the burned clinker must be reduced to a powder to enable mixing, placing, and hardening of the cement and water. While the evolution of early fineness testing is obscure, it is apparent that with the knowledge of the powder characteristic requirement of cementitious materials, man in his quest for more knowledge began to be concerned with the extent of grinding required to produce the best performing powder. Costs of grinding were, as now, of importance in the early days. Ground on mill stones in much the fashion of grains, opposition developed back in 1881 to Faija's proposals that "the importance of fine grinding cannot be overestimated" and the manufacturers responded with complaints of "the expense of fine grinding—wear and tear of millstones, etc."

Screen Test Procedures:

The earliest screenings were made by means of bolting cloth, a coarse open-weave cloth with no standard dimension of opening but sometimes classed by warp and woof count of threads per inch. Such screening, borrowed from the grain milling industry, merely served to remove from the cement unground particles which were frequently discarded.

Since the reject portion represented appreciable amounts of the cement clinker, studies were instituted to evaluate this material. In the mid 1800's it was found that particles retained on the screen, if reground, developed greater strengths than the previously collected fine portion, and that this reground portion was slower setting than the original fine portion. Since this work preceded the use of gypsum for set stabilization, an obvious advantage was gained by regrinding the previously discarded screen rejects and adding the pulverized material to the original fine product. It was a simple matter of observation to deduce that the previously rejected particles represented more thoroughly burned or sintered material; this led to direct attempts to measure degree of burning by visual observation and subsequently the liter-weight test.

Such findings led rapidly to development of specific screen dimensions, and before the turn of the century screens of 200 mesh or 40,000 openings per square inch had been devised of woven wire.

Woven wire screens continued to be developed and in 1925 the first ASTM Tentative Specification for, Sieves for Testing Purposes, was issued, followed by a separate ASTM Standard Specification for, Round Hole Screens for Testing Purposes, (E 17)[4] a few years later.

The current Specifications E 11 covers both woven wire and round-hole screens ranging from 8 in. openings for the round-hole plate screen to the No. 400 sieve designated as 37-μ opening with 71 separate screen or sieve designations, each with a specified tolerance of opening size, wire or plate dimension, and types and finish of allowable metals used. Here it is of interest to note that Specification E 11, Section 2(*b*), states that standard sieves shall be woven from brass, bronze, or other suitable wire, whereas Specification C 430, Fineness of Hydraulic Cement by the No. 325 Sieve,[5] in Section 2(*a*) requires a stainless steel woven wire screen cloth. Thus after over 100 years of screen development, studies still continue to refine this test precedure.

In 1960 the Tentative Specification for Precision Micromesh Sieves[6] (E 161), was issued. These sieves are square-hole electroformed micromesh formed by electrodeposition of copper or nickel in a firm crystalline structure on a flat supporting copper grid. Nominal opening size ranges from 149 μ to 5.5 μ. Their use is restricted to calibrations of normal woven wire screens and research studies of particle size and distribution.

In a recent unpublished work, O. E. Brown of Marquette Cement Manufacturing Co. made a study of the characteristics of the bronze No. 325 sieve, the stainless steel No. 325 sieve, and the 44-μ precision micromesh sieve. The results of his study have been made available to the Working Committee on Fineness of ASTM Committee C-1 on Cement. His study revealed that the standard No. 325 sieve of bronze wire with its nominal 44-μ opening ranged from less than 40 μ to an average of 50 μ and a maximum of 60 μ. His work further showed an appreciably closer tolerance available with the stainless steel No. 325 sieve currently specified in Specifications C 430.

History has an odd habit of repeating itself. While it is probable that almost no mix control chemist and very few senior scientists appreciate the fact or its reasons, the current Method C 115[7] reflects this same fact. Originally published by Wagner in 1933 (2), the description, test procedure, and calculation correct for the percentage of sample passing the No. 325 (44 μ) sieve, and yet the first recorded reading of the suspension of cement known as I60 corresponds to a 60-μ particle diameter.

In the original study of this test procedure Wagner ran repeated tests on the No. 325 sieve procedure and microscopically evaluated the particle size passing and retained on this sieve. After literally hundreds of such examinations he concluded that particles finer than 60 μ passed and only particles larger than 60 μ were retained on the 44-μ sieve. Since the diagonal of the 44-μ square opening is approximately 60 μ he decided that this fact accounted for the retention of the screen and subsequently established the entire surface area calculation on this premise. Brown's current work lends further credence to the premise that the suspension turbidity should include 60-μ material measurements based upon both the diagonal of the opening and its inaccuracy.

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[4] These two standards were subsequently combined in Specifications for Sieves for Testing Purposes (Wire Cloth Sieves, Round-Hole and Square-Hole Plate Screens or Sieves) (E 11), 1961 Book of ASTM Standards, Parts 3, 4, 5, and 8.

[5] 1961 Book of ASTM Standards, Part 4.

[6] Specifications for Precision Micromesh Sieves (E 161), 1961 Book of ASTM Standards, Parts 3, 4, 7, and 8.

[7] Method of Test for Fineness of Portland Cement by the Turbidimeter (C 115), 1961 Book of ASTM Standards Part 4.

The Microscope:

With the advent of finer grinding, limitations of screen testing were noted. With greater percentages passing the finest sieves available, less knowledge was obtainable concerning a greater portion of the material. With current cements passing 80 to 100 per cent through a No. 325 sieve, knowledge of the characteristic of the 80 to 100 per cent of the material was limited to the fact that it was less than 44 or, more properly, 60 μ. There was no information available on distribution of particles less than 60 μ nor on the extent of fineness attained on the finest particles.

The use of optical microscopy extended the range of study below the 60-μ limit to less than a single micron, with the additional knowledge of particle shape and surface characteristics. At this limit the distribution, shape, and surface characteristics of cement could now cover approximately 95 per cent of the entire sample, leaving only the 5 per cent or less of submicron material as an unknown.

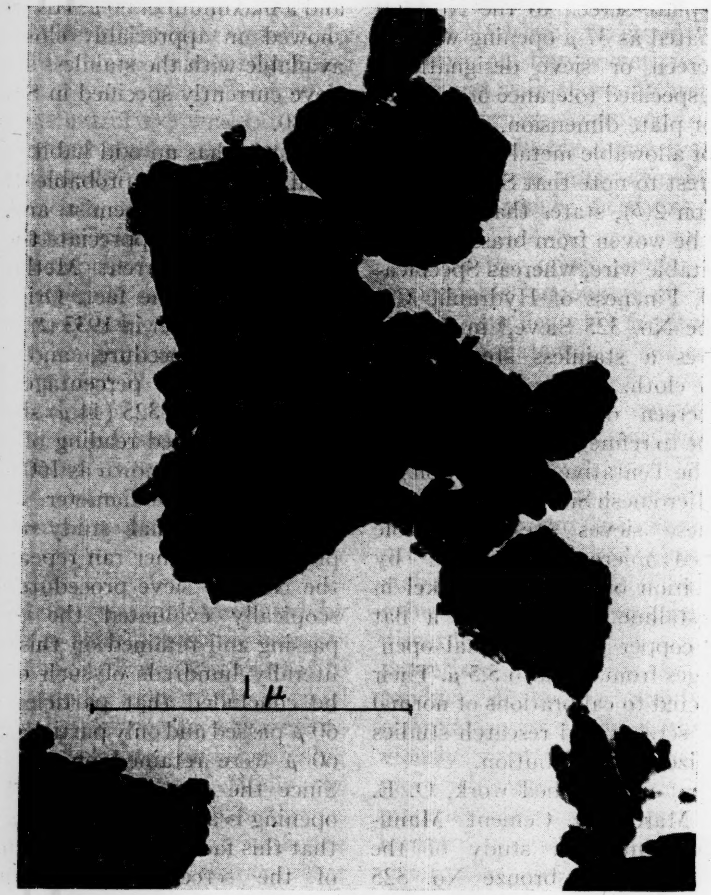

FIG. 1.—Electron Micrograph of Fine Fraction Type I Cement.

By the use of stage or ocular micromeasuring grids and the tedious task of counting, comparing, and tabulating hundreds of individual particles an accurate particle size distribution may be obtained. The facility is improved somewhat by photomicrography techniques, by projection microscopes, and even some three-dimensional characteristics may be observed with binocular microscopes, although these are limited in magnification power to resolution of about 5 to 10 μ.

Limitations on the use of microscopic techniques involve the small samples that must be used; for example, on oil immersion lens systems required for resolution under 1 μ the actual sample under observation is in the order of 0.001 g. Many fields must be examined, counted, sized, and calculated back to weight percentage to ensure any accuracy—100 fields of optical microscopy for each 0.1 g of cement. Fatigue and time also seriously limit the use of microscopic techniques. If screen fractions are determined at No. 200, 325, and No. 400-sieves, a minimum of three microscopic fields at each fraction are required with a minimum count of 250 particles in each field of the −No. 400 sample. It is obvious that this technique cannot become a control tool nor a routine method of fineness evaluation. Its use is primarily limited to calibration of other and faster procedures as, for example, the turbidimeter, or to cases where materials cannot be assumed to follow Stokes' law in an approximate manner.

ASTM Recommended Practice E 20[8] gives an excellent background for this type of analysis and includes an extensive reference list for those interested in further study.

The microscopic technique is probably the most accurate of all particle size study systems, and yet its limitations are readily apparent. The advent of the electron microscope has extended the range to the submicron fields, the less than 5 per cent unknown material, although with further restrictions on size of sample and an increase in the required number of fields examined. Figure 1 is an electron micrograph of the finest fraction separated from a type I portland cement taken by L. E. Copeland (Portland Cement Assn., Research and Development Laboratories) and gives new insights into the submicron characteristics of cement.

Stokes' Law (3):

Stokes' law has been the basis for many particulate analysis schemes both by direct and by indirect measurement of the rate of settling of particles in a fluid medium, either liquid or gaseous. Stated nonmathematically, Stokes' law states "when a spherical particle is left undisturbed in a medium in a gravitational field, it will at first sink with increasing velocity, but when the resistance is equal to the acting force it will reach a constant velocity." Mathematically the relationship must include values of density of the particle and the medium of suspension, acceleration due to gravity, radius of the particle, velocity of the particle, and viscosity of the medium. When applied to the determination of the particle size of materials such as cement it is obvious that the assumption of a sphere does not literally apply. Here comparison of calculated values from sedimentation tests may be applied against microscopic analysis for establishment of appropriate corrective constants to make application of Stokes' law relatively valid.

A brief statement of each of the methods used which involve Stokes' law indicates the wide uses to which the principle has been applied.

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[8] Recommended Practice for Analysis by Microscopical Methods for Particle Size Distribution of Particulate Substances of Subsieve Sizes (E 20), 1962 Supplement to Book of ASTM Standards, Parts 4 and 8.

1. Cumulative measurement of total amount of material in suspension left above a certain level. In this procedure a column of suspension is balanced against a manometric column of clear, nonmiscible liquid. The change in level of the manometer with time provides a very simple means of following the sedimentation.

2. Direct measurement of weight of material settled below a given horizontal plane. Numerous balance methods weighing the settled material at the base of a settling column have been devised. The most publicized method, applied in limited cases in the cement industry, is represented by the Sharples Micromerograph which uses air as the fluid medium.

3. Measurement of particle concentration at a particle level and time increment. Four variations of this application have appeared in the field of particulate analysis:

(*a*) The differential manometer, a refinement of method 1 above, but sensitive to minor temperature shifts.

(*b*) The hydrometer method as exemplified by ASTM Method for Grain-Size Analysis of Soils (D 422),[9] has found wide application for approximate particle size analysis with many variations in hydrometer design and techniques.

(*c*) The pipet method, commonly known as the Andreasen pipet, withdraws samples from a settling column at time intervals selected from calculations to represent various micron fractions. The method is one of the most widely used of all sedimentation tests although rarely applied to portland cement.

(*d*) The photoextinction methods of following sedimentation are exemplified in the cement industry by the Wagner turbidimeter. Having the major advantage of not disturbing the sedimentation column but merely relating the decrease in turbidity with the decrease in mass concentration gives a rapid reproducible test procedure.

4. Direct photographic systems have been tried where the tracks of individual particles while settling are directly recorded on a photographic plate. Sizes are estimated from the length of the track. This procedure, even more than the microscope measuring technique, requires tedious measurement of many tracks to arrive at a statistically accurate number.

5. Decantation methods allow particles to settle from the suspension for precalculated periods of time to relate to a given micron size range; the supernatant liquor is decanted and the residue dried and weighed.

6. Centrifugal systems relate rather closely to decantation methods but offer the advantage of lowering the limit of particle size that can be measured by substituting centrifugal for gravitational acceleration.

7. Elutriation is a reversed application of Stokes' law in which the suspending medium is kept in a state of motion and depending upon the rate of motion, particles of predetermined size are kept in suspension. In the cement industry some application has been made of air elutriation as exemplified by the Roller air sizer.

Permeability Methods:

Frequently, the total surface area of a powdered material gives an indication sufficient for control or comparison purposes to eliminate the need for studies of particle size distribution with attendant time and calculation requirements. This method is exemplified by the Blaine air permeability test. ASTM Method C

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[9] 1961 Book of ASTM Standards, Part 4.

204[10] results in a single total surface area value. The number and size of the pores in a prepared bed of sample of definite porosity is a function of the size of the particles and determines the rate of fluid flow through the bed.

The Blaine apparatus, based on earlier work by Carmen on liquid permeability and the 1939 experimental work and development of Lea and Nurse on powdered materials, represents a simple rapid relative measurement of this pore spacing in properly compacted cement samples. The Blaine apparatus is universally used in cement laboratories as a control tool for measurement of grinding results.

Other Procedures:

Of limited application in the cement industry and used primarily in research studies on particle size, shape, and characteristics are a group of liquid or gas adsorption techniques based upon adsorption of a monomolecular layer of either gas or liquid. These procedures evolve values of total surface including the areas of cracks, crevices, and surface distortions down to molecular proportions as contrasted to the measurement of the external visible dimensions of the particles attained by methods previously described.

Electronic size analysis described by Berg (4) is the newest procedure to invade the field of particulate analysis. The technique is based on the Coulter principle and consists of forcing a suspension of particles in a conductive liquid through a small aperture having an immersed electrode on each side. As each particle passes the aperture it replaces its own volume of electrolyte within the aperture, momentarily changing the resistance value between the electrodes. This change in resistance is noted as a voltage pulse of time duration of passage. The pulse signal is amplified and indicated on a pulse counter. By using more than one aperture size (sizes now in use range from 10 to 1000 μ) and with the wide range of particles that may pass through and influence resistance proportionally (estimated at 20:1) for any one aperture, the range and reproducibility of results from this apparatus open new horizons for the study of particle size phenomena. Currently some research work is being conducted in the cement industry with this tool.

In any of the test procedures—from simple screen tests to the electronic size analysis—all procedures have inherent in their makeup certain assumptions such as the spherical shape of particles assumed for Stokes' law applications to adequacy of dispersion in the Coulter principle. The use of assumptions does not preclude the usefulness of the procedure and results, while not capable of being absolute values, do serve as reproducible relative numbers.

That particle size and its influence on properties of materials is significant is attested by the fact that in ASTM alone there are 45 separate specific standards for particle size measurement and literally hundreds of material specifications contain additional test procedures and fineness requirements.

SIGNIFICANCE OF FINENESS TEST RESULTS

Fineness itself is not a property which the consumer evaluates except as he might seek compliance to all items of a product specification. The consumer is concerned with strength, rate of strength development, set time, soundness constancy of volume, bleeding, curing characteristics, air content, heat of hydration, false set, reaction with aggregates, and workability. Some, but by no means all of these performance specifications are

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[10] Method of Test for Fineness of Portland Cement by Air Permeability Apparatus (C 204), 1961 Book of ASTM Standards, Part 4.

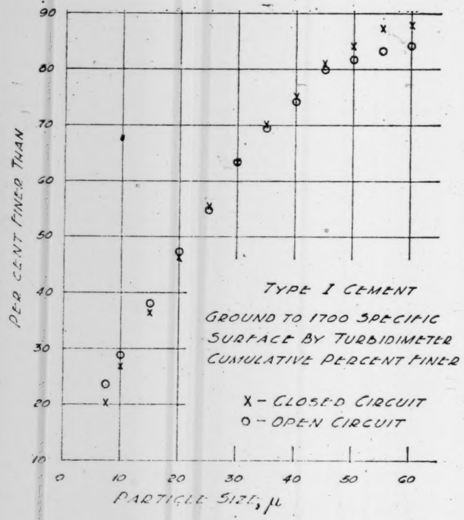

FIG. 2.—Effect of Closed and Open Circuit Grinding on Fineness Distribution.

TABLE 1.—TENSILE STRENGTH OF CEMENT AS-RECEIVED AND AFTER ADDITIONAL GRINDING.

Cement	Tensile Strength, psi	
	7 days	28 days
No. 1:		
As-received	535	661
Ground passing 50 mesh	572	...
No. 2:		
As-received	509	650
Ground passing 50 mesh	542	675
No. 3:		
As-received	481	650
Ground passing 50 mesh	505	710

currently part of ASTM Specifications C 150. The consumer's concern with portland cement is in performance and his reliance on fineness specification is a guide to anticipated performance.

The producer on the other hand, is concerned with fineness for many reasons. The rapidity with which the fineness tests can be completed and their known influence on performance properties of portland cement make such tests among the most facile control tools available to the manufacturer.

It can be readily demonstrated that fineness has significant influence on performance properties. Unfortunately the influence of raw material quality, its preparation, the blending of raw materials, the uniformity of compound composition, the preheating, calcining, and sintering of the cement clinker, its potential cement compound composition, the quenching of the clinker, the sulfur trioxide content of the cement from its final grinding, the use of grinding aids or processing additions—all these plus additional factors also influence the performance of the cement in concrete, some to a greater degree than fineness of the cement. Certainly manufacturing costs bear a direct relationship to fineness—finer grinding requiring additional power.

TABLE 2.—STRENGTH INCREASE BY REASON OF FINENESS.[a]

	Cement 1	Cement 2
No. 325 fineness, per cent	88.0	99.0
Wagner specific surface, sq cm per g	1765	2830
Blaine surface area, sq cm per g	3030	5059
Compressive strength, psi:		
3 day	2580	4570
7 day	3860	6430
28 day	5590	7035
Tensile strength, psi:		
3 day	342	453
7 day	423	485
28 day	504	540

[a] The clinker and gypsum type and amount were the same for both grinds, the cement being essentially a moderate C_3S and relatively high (10 per cent) C_3A product.

Open Versus Closed Circuit Grinding:

Open versus closed circuit grinding is presumed to affect the performance characteristics of portland cement although many examples exist of interchange of open versus closed circuit cement on concrete with no detectable changes. In the industry today most cement is produced in closed circuit systems using air separators, though several examples of open circuit grinding still exist in the field.

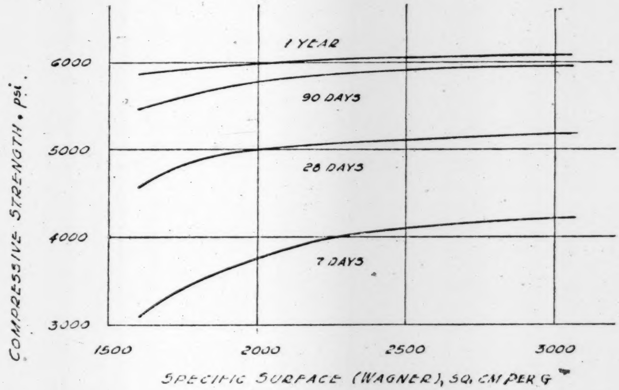

FIG. 3.—Effect of Fineness of Cement on Strength of Concrete.

TABLE 3.—RELATIONSHIP BETWEEN STRENGTH, FINENESS, AND CURING.

	70 F Moist Cure			5½ hr 70 F Moist Cure, 140 F Steam Cure	
	1 day	3 day	7 day	5 hr	16 hr
Cement No. 1, 2960 Blaine surface area	610	1550	2260	510	1595
Cement No. 2, 3600 Blaine surface area	1395	2900	3800	1235	2405

	24 hr 70 F Moist Cure, 7½ hr 150 F Steam	24 hr 70 F Moist Cure, 3 hr Autoclave, 300 psi, 430 F
Cement No. 1, 2960 Blaine surface area	1365	4525
Cement No. 2, 3600 Blaine surface area	2485	4700

Theoretically open circuit grinding will result in a normal distribution curve of particles from coarsest to finest, depending upon the maximum size particle allowed to leave the system; a closed circuit system will, by removal and return of the coarse particles to the mill and by passing through those particles already sufficiently fine for cement uses, produce a much narrower range of particles with no representative particles of large sizes and reduced numbers of ultrafine particles. If a theoretically perfect closed circuit system were devised, all particles removed as product would be of a single size.

In practice, however, this has been difficult to establish to any degree of certainty. The operation of air separators as practiced in the United States cement industry with relatively high circulating loads and high mill output rates is actually an inefficient operation. Much of the circulating load is already of fineness suitable for cement in concrete and frequently oversize spitzers are found in the separator product.

Comparison of fineness between open circuit and closed circuit grinding using the same two-stage mill system, the same cement clinker, and successive grinds with and without air separation showed negligible differences in particle size distribution. Figure 2 shows the cumulative percentage curve obtained on these two trial grinds on commercial equipment. While the curves are based on turbidimetric results which did not actually measure below 7.5 μ, it does not appear logical that such differences as are exhibited between open and closed circuit grinding in this test are significant.

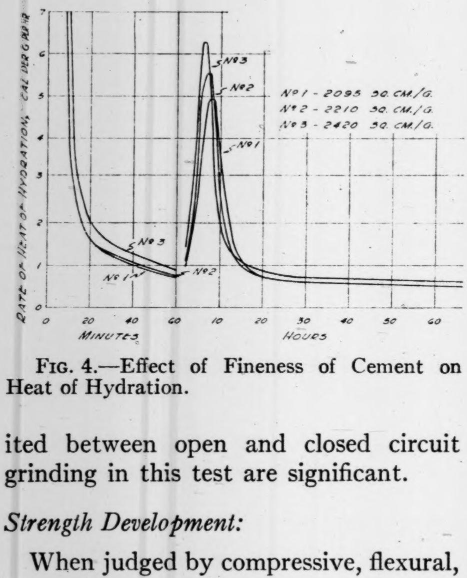

FIG. 4.—Effect of Fineness of Cement on Heat of Hydration.

Strength Development:

When judged by compressive, flexural, or tensile strength at a given period of time there can be no doubt that fineness of a given clinker grind is significant in the strength attained. Table 1, taken from *Proceedings*, Royal Institute British Architects, 1879–80, p. 113 (1) shows results of tests made on cement as-received and as ground additionally to pass a 50-mesh sieve.

In a modern day recheck of this increase in strength obtained with further grinding samples of clinker ground in a commercial mill at prevailing finenesses, the data shown in Table 2 indicate the same trend although in the present case the difference is of greater magnitude owing to the much greater increase in surface area.

Lea (5) states that the more finely a cement is ground the greater is its strength. Figure 3, taken from his text, supports this statement.

Rate of Strength Development:

Careful examination of Fig. 3 shows that the major influence of fine grinding lies in the rate of strength development rather than in the actual increase of strength in ultimate or near ultimate hydration. If one will extrapolate beyond the one-year line of this graph, it seems reasonable to predict that future curves of this same cement would result in an almost constant strength value despite fineness.

In an experiment of rate of strength development, two cements identical in composition were cured in moist conditions, 140 F steam chambers, 150 F steam chambers, and 300 psi 430 F autoclave conditions. The difference in these cements lay in their fineness. Table 3 gives the comparative data.

Examination of the data in Table 3 will disclose that a cement with 2960 air permeability surface area lags well behind an equivalent cement with 3600 surface area in all strength development conditions, except the 3-hr autoclave period where the difference is less than 4 per cent in total strength as compared to a difference in excess of 100 per cent at 1-day moist cure.

The relatively high tensile strengths shown in Table 1 as compared to the normal present-day tensile strength of Table 2 when it is obvious that the cements of Table 2 are much finer than those of Table 1 are open to some comment. This apparent discrepancy is not basically related to the strength-gaining properties of the cement iteslf. The high tensile strengths in Table 1 may be duplicated with modern cements if tension briquets are made with 3 parts of aggregate to 1 part of cement, if the aggregate used is partially ground, 30–50 mesh, portland-cement clinker. The bond strength of cement paste to clinker sand sized aggregate is far in excess of the bond strength of cement paste to normal siliceous or limestone sands. This extreme bond results from hydration reaction products between the cement paste and the surface of the clinker sands and results in abnormally high tensile strength.

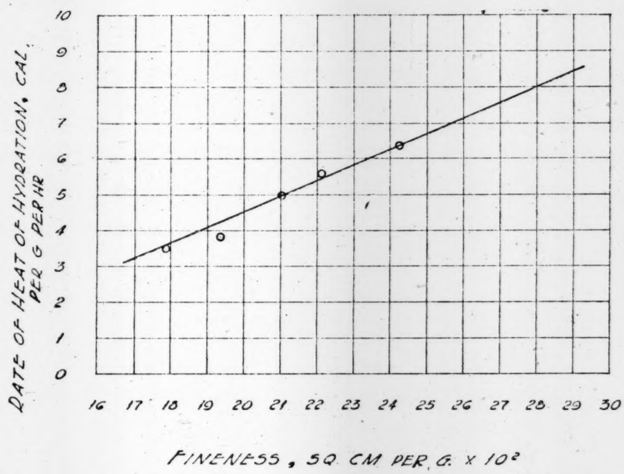

FIG. 5.—Effect of Fineness of Cement on Maximum Rate of Hydration Versus Surface Area.

Heat of Hydration:

Directly related to the rate of strength development is the rate of development of heat of hydration. A peculiar consumer attitude has arisen which does not appear to be aware of this correlation. A current practice is to specify moderate heat of hydration cement with heat of hydration judged solely by a 58 per cent limitation on combined C_3S and C_3A and then to further specify 3- or 7-day strength levels which make mandatory high fineness cements. The increase in fineness largely negates the moderate heat of hydration limits considered available by the compound composition limits. Figure 4 shows the heats of hydration of equivalent cements with difference in fineness being the contributing factor in the heat of hydration curves.

Similar data have been plotted in Fig. 5 on five equivalent cements for maximum rate of hydration versus surface areas by turbidimetric test from 1780 sq cm per g to 2420 sq cm per g.

Tests have not been conducted relating this factor to sulfate resistance of cements which may be governed by compound composition and not by rate of strength development or rate of heat of hydration development. It is suggested, however, that before relying on this assumption, specifications for the use of sulfate-resistant cements should at least be aware of the changes produced in properties of cement by fineness.

Workability:

Since the measurement of workability

has defied numerical evaluation, no positive relationship between fineness and workability has been determined. The amount of water required to produce a neat cement paste of normal consistency increases with increasing fineness of the cement. As the fineness increases, however, the water requirement for a constant slump concrete decreases until the higher ranges of type III fineness are reached. Lea (5) predicted that if cements were produced at 7 μ or less, almost completely unworkable concretes would result from the sticky cohesive nature of such material when mixed with water.

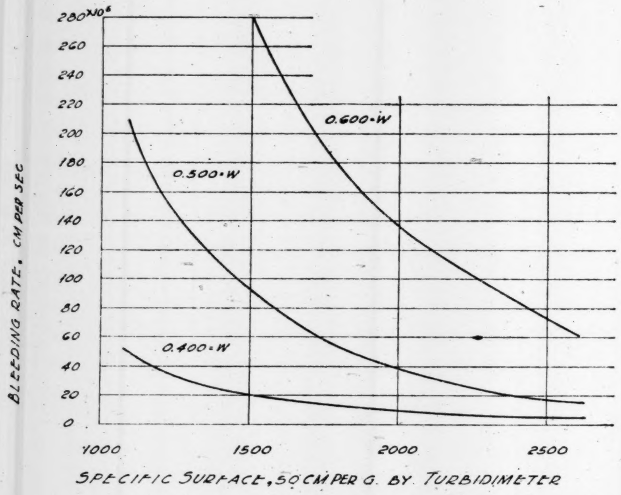

FIG. 6.—Effect of Fineness and Water Content on Bleeding of Concrete Paste.

If water requirements are increased by reason of the fineness and the type of concrete or cement product mix used, it is also known that the drying shrinkage of the resultant set concrete or concrete product will be increased proportionally.

Bleeding:

The influence of fineness on bleeding is described by Powers (6) and further refined by Steinour (7). These papers conclude that specific surface of the cement is the most important factor controlling the bleeding characteristics of a portland cement. However, these papers continue to point out other factors that govern bleeding rate to the extent that fineness alone cannot be used as a predictive basis for probable bleeding characteristics. Listed as of influence are the particle shapes, the state of flocculation on mixing, the extent and type of early reactions on mixing, the water-cement ratio, the gradation of aggregates and their shape, the alkali content, the tricalcium aluminate content, the clinker quenching, the gypsum addition, the temperature of grinding, the use of air-entraining agents, calcium chloride acceleration, and aeration of the cement. In the words of Powers "One point made clear by the extended studies is that there is no hope of finding a universal simple formula for predicting bleeding characteristics of a cement from its composition." Figure 6 taken from Steinour's data shows the effect of cement fineness at three water contents; it may be seen that higher finenesses and lower water-cement ratios reduce bleeding in major amounts.

In view of the large number of known

factors and presumably some unknown factors governing bleeding characteristics of portland cement, it is the author's opinion that the bleeding test itself would be a more protective specification than increasing cement fineness as a cure-all. ASTM Method C 243,[11] intended solely for use in research, might well be used as a performance specification by limiting the time of test to early periods and by simplification of test procedure and apparatus.

SUMMARY

It may be concluded that the fineness of portland cement is of significance and does influence to major degrees many of the performance properties of cement in concrete or concrete products.

Fineness is most properly a manufacturing control tool seeking to maintain uniformity of product and constant performance characteristics.

While the consumers' interest in fineness may give him valid knowledge regarding the cement, caution is advised in seeking either excessively high or low fineness cements for special application without thorough study and evaluation of the influence of the sought-after fineness on properties other than rate of strength development, the commonest criterion for cement quality.

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(2) L. A. Wagner, "A Rapid Method for the Determination of the Specific Surface of Portland Cement," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, 1933.

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(4) R. H. Berg, "Electronic Size Analysis of Subsieve Particles by Flowing Through a Small Liquid Resistor," Symposium on Particle Size Measurement, *ASTM STP No. 234*, Am. Soc. Testing Mats., 1958, p. 245.

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[11] Method of Test for Bleeding of Cement Pastes and Mortars (C 243), 1962 Supplement to Book of ASTM Standards, Part 4.

SIGNIFICANCE OF FALSE SET TESTS*

By J. L. Gilliland[1]

Synopsis

ASTM has adopted optional specification limits for false set in portland cement that are not in effect unless specifically requested by the purchaser. Inasmuch as false set is of little concern in many portland cement applications, it is recommended that the specific need for the limitation should be considered prior to the inclusion of the option in the purchase order.

The specification limitation is based on the paste test method, which is similar to the procedure that has been a part of the Federal specification for many years.

A mortar test method is also available, and is recommended for research studies.

Because of the effect of aeration on false set, samples on which the test is to be made should be protected from exposure.

At the 1961 Annual meeting of the Society, W. C. Hansen[2] presented a discussion of the tentative methods of test for false set in portland cement. His report was concerned particularly with the studies which preceded and followed the adoption in 1955 of the mortar test method C 359.[3] Features of the Nebraska Highway Dept. paste test and the Washington Highway Dept. mortar test were combined into this new mortar method.

The ASTM paste test method C 451,[4] adopted in 1960, was patterned closely after the method that has been a part of the Federal specification for many years.

To understand the significance of these tests, it is important to recognize the basic difference between them, and the unusual character of the property called false set. The basic difference between the methods is in the amount of water used in preparing the mortar or paste. In the mortar method, C 359, the water is specified as 30 per cent by weight of the cement, except that 32 per cent water is used for type III cements. In the paste method, C 451, the water is varied to give an initial penetration of 34 ± 4 mm.

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* Presented at the Sixty-sixth Annual Meeting of the Society, Atlantic City, N. J., June 23–28, 1963.

[1] General Chemist, Ideal Cement Co., Denver, Colo.

[2] W. C. Hansen, "Discussion of Tentative Methods of Tests for False Set of Portland Cement," *Proceedings*, Am. Soc. Testing Mats., Vol. 61, p. 1029 (1961).

[3] Method of Test for False Set of Portland Cement (Mortar Method) (C 359), 1961 Book of ASTM Standards, Part 4.

[4] Method of Test for False Set of Portland Cement (Paste Method), (C 451), 1961 Book of ASTM Standards, Part 4.

COMPARISON OF THE TEST METHODS

The use of a fixed water-cement ratio in Method C 359 is a definite disadvantage to any cement that, for one reason or another, has a high water requirement. Although the method recognizes the higher water requirements of type III cements, it has no compensating factor for cements of type I or II which may be somewhat finer than normal. Some cements will not give an initial penetration of 50+ at 30 per cent water, and therefore show low penetrations at the subsequent time periods. But when these same cements are tested at 31 or 32 per cent water, penetrations of 50+ are obtained at all test periods. The Washington method used the flow table to establish the water content of the mortar, but the Working Committee on False Set of ASTM Committee C-1 on Cement felt that this simply introduced another variable.

On the other hand, objection has also been made to the variable water content permitted by Method C 451 because, with hand mixing, the water requirement is a function of the skill and vigor of the laboratory technician. Establishing the water requirement by this method is especially difficult for cements having severe false set. The latest revision, C 451 - 62 T,[5] provides for mechanical mixing of the paste to minimize the human factor in establishing the water requirement.

SPECIFICATION LIMITS

Although no limits have been adopted by ASTM using Method C 359, Hansen reported: "It appears from these results that cements that cause difficulties in the field from the standpoint of false set are likely to have penetrations of less than 5 mm at the 11-min period."

The Michigan State Highway Dept.[6] has adopted the mortar method in its "Supplemental Specifications for Portland Cement, April 18, 1962." Its requirement is: "Portland cement shall not develop false set when tested in accordance with the current Method of Test for False Set of Portland Cement (Mortar Method), ASTM Designation: C 359. False set will be considered to have occurred if the penetration is less than 5 mm at the 5, 8, or 11-min intervals as defined in the test method."

At the June 1962 meeting of the Working Committee on False Set, it was reported that the Mexican Bureau of Reclamation uses Method C 359, specifying a limit at 11 min of not less than 50 per cent of the initial penetration.

The Hydro-Electric Power Commission of Ontario,[7] in purchasing cement for the Little Long generating station project on the Mattagami River, has included the following requirement in their purchase orders:

> The setting properties of the cement shall be such that when tested according to ASTM Method C 359, the cement shall not show false or flash set. Such tests shall be made on mortar conforming not only to the designated temperature range, but also on mortar having a temperature as low as 50 F. A penetration of the designated needle of less than 5 mm in 11 min or less shall be considered false or flash set.

The Working Committee on False Set has frequently considered the question of specifications limits for the mortar method but has not recommended the adoption of any limits because of the lack of acceptable correlation with the field performance of concrete. However, the method has been retained because of its value to research workers.

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[5] 1962 Supplement to Book of ASTM Standards, Part 4.

[6] Michigan State Highway Dept. Supplemental Specifications for Portland Cement, April 18, 1962.

[7] The Hydro-Electric Power Commission of Ontario, personal communication from W. E. Parker, Toronto, April 30, 1963.

The paste test has had many years of specification application as a part of Federal Specification SS-C-192. The ASTM paste method uses a wider range of initial penetration than does the Federal method in establishing the water requirement. This change is of particular help when testing cements with severe false set. Test limits have been approved by Committee C-1 as optional requirements for Specifications C 150[8] and C 175,[9] specifying that the final penetration shall be not less than 50 per cent of the initial penetration. It is anticipated that the next revision of Federal Test Method Standard No. 158[10] will incorporate machine mixing and initial penetrations of 34 ± 4 mm, in conformance with Method C 451. The 50 per cent limit on the final penetration has been adopted in Interim Federal Specification SS-C-00192e.[11]

DISCUSSION ON THE TEST METHODS

The ASTM Methods C 359 and C 451 include the following definitions and interpretation:

Definitions

2. (*a*) *False Set.*—False set is the rapid development of rigidity in a mixed portland-cement paste, mortar, or concrete caused by the crystallization of gypsum throughout the cement paste. This rigidity may vary from mild to severe. It will develop without the evolution of much heat and can be dispelled, usually with almost complete restoration of the plasticity, by further mixing without addition of water.

(*b*) *Quick or Flash Set.*—Quick or flash set is the rapid development of rigidity in a mixed portland-cement paste, mortar, or concrete caused by the rapid reactions of certain of the cement minerals with water. Quick set may vary from mild to severe, depending upon the degree to which the rates of these reactions have been controlled by treatment of the clinker and the addition of untreated calcium sulfate during grinding. Heat is evolved during these reactions, and with severe quick set the temperature of the mass rises very rapidly. Further mixing without the addition of water will destroy this rigidity if the quick set is relatively mild, but not when the quick set is severe.

Interpretation

7. (*a*) Severe false set tendencies in a cement may cause difficulty, from a placing and handling standpoint, but it is not likely to cause difficulties where concrete is mixed for a longer time than usual, such as usually occurs in transit mixing, or where it is remixed prior to placing or transporting, such as occurs in concrete pumping operations. It is most apt to be noticeable where concrete is mixed for a short period of time in stationary mixers and transported to the forms in nonagitating equipment, such as on some paving jobs.

(*b*) False set, per se, has no deleterious effects on the quality of concrete; however, cements with severe false setting tendencies usually require slightly more mixing water to produce the same consistency, which may be expected to result in slightly lower strengths and increased drying shrinkage.

(*c*) Quick set tendencies of a severity sufficient to cause difficulties, from a placing and handling standpoint, usually will cause the cement to fail the requirements for time of set in Standard Specifications for Portland Cement, ASTM Designation C 150.

It is to be noted that the distinguishing features between false set and quick set are the degree of heat evolution and the response to remixing. While severe quick set should be readily identifiable by means of the heat evolution, mild quick set can be mistaken for false set and vice versa. The mortar test provides a remixing procedure to distinguish between quick and false set. While remixing is not specified in the paste method, the investigator may wish to remix the paste to verify that the plasticity can be restored when a particular cement is under study. The committee has felt, however, that it is unnecessary to include remixing as a specification requirement since the loss in plasticity is the objectional feature, regardless of whether it is due to quick or to false set.

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[8] Specifications for Portland Cement (C 150), see 1963 Report of Committee C-1 on Cement.

[9] Specifications for Air-Entraining Portland Cement (C 175), see 1963 Report of Committee C-1 on Cement.

[10] Federal Test Method Standard No. 158a, Sept. 23, 1960; and amendment draft of Aug. 27, 1962.

[11] Interim Federal Specification SS-C-00192e (March 25, 1963).

It is not the function of this paper to review the theories on the causes and mechanisms of false set in portland cement. The serious student of the problem is referred to the recent paper presented by W. C. Hansen[12] at the Fourth International Symposium on the Chemistry of Cement. The discussions accompanying Hansen's paper show the world-wide interest in the problem. Briefly, it is generally agreed that the dehydration of gypsum, during grinding or storage, is the principal cause of false set in commercial portland cements. As noted in the above definition, the crystallization of this dehydrated gypsum imparts the rigidity to the cement paste.

It seems probable that the gypsum is dehydrated to some degree in all commercially ground portland cements. The actual extent of dehydration is a function of temperature, time, and relative humidity of the grinding, transporting, and storing operations.

PRECAUTIONS IN USING THE METHODS

The presence of some dehydrated gypsum does not necessarily impart abnormal setting behavior to the cement or to the concrete in which it is mixed. The degree of actual false set is a function of: (1) the rate of soluble sulfate availability in the paste, (2) the sulfate demands of the paste, (3) the physical manipulation of the paste, and (4) the degree of saturation of the mixing water for calcium sulfate dihydrate.

If the rate of availability is less than or equal to the sulfate demands, false set will not occur because the sulfate is consumed as rapidly as it appears in solution; therefore a condition of supersaturation is not attained and gypsum has no opportunity to crystallize. If the rate of availability is considerably less than the demands, insufficient sulfate will be available, and quick set may result.

Hemihydrate and soluble anhydrite dissolve rapidly and form solutions that are supersaturated with respect to gypsum if the sulfate is not rapidly consumed. The equilibrium solubility, however, is a function of the soluble alkalies as well as the solution temperature; therefore both factors must be considered in judging supersaturation. False set may be anticipated if the degree of supersaturation is great (after allowing for the effect of the alkalies).

Prolonged mixing or manipulation of the wet mass will break up the tendency to false set and can even overcome mild cases of quick set.

Both the sulfate solubility and the sulfate demands of the paste are affected by aeration; thus it is important to minimize the exposure of the cement sample. Storage in a paper sack is a poor practice. Any sample that has not been shipped in a plastic or metal container is of questionable value and should not be used to evaluate a shipment or stock of cement.

Finally, it is important to consider the manner in which the cement will be used. Transit mixers usually agitate for sufficient time to destroy any tendency to false set. Concrete-pumping installations include a remixing operation that serves the same purpose. In these cases, even a severely false-setting cement may not be a problem.

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[12] W. C. Hansen, "False Set in Portland Cement," Chemistry of Cement, Nat. Bureau of Standards, *Monograph 43*, Vol. 1, p. 387 (1962).

On the other hand, as noted previously in this paper, a critical situation may be encountered on paving jobs where low water-cement ratio concrete is mixed for a very short time, then transported to the forms without agitation, or allowed to be quiescent for a brief period before leveling.

Since some dehydrated gypsum exists in all commercial portland cements, it seems equally accurate to say that some false set potential exists in all commercial portland-cement pastes, mortars, and concretes. The major problem is to establish a specification method suitable for identifying those cements that would be expected to give trouble in the field. In Method C 451, the paste mixing period totals 3 min. The committee recognizes that the test can be made more severe if the mixing period is shortened. During the cooperative study prior to the adoption of Method C 451, a range of mixing times from 30 to 240 sec was used. It was concluded that cements with only slight false set might be rejected if the time were made too short. On the other extreme, cements having severe false set are identified after any reasonable mixing time. As is always the case, the intermediate cements are the most difficult to classify, but the committee consensus was that the 3-min (total) mixing time was the most satisfactory.

At the time of the cooperative study, the laboratory of the United States Bureau of Reclamation reported the results shown in Table I. The report was accompanied by this amplification:

> "More than a ton of each (cements D and E) have been used in many laboratory concrete mixes over several months' time without exhibiting false set. However, these two cements would be classified as false-setting for mixing times of about 120 sec or less."

TABLE I.—EFFECTS OF MIXING TIME ON CEMENTS (HAND MIXING OF PASTES)

Mixing Time, sec	Final Penetration, per cent, for Cements							
	A	B	C	D	E	F	G	H
30	73	48	40	9	0	0	0	0
60	...	...	...	25	...	...	0	...
90	...	...	...	46	...	...	0	...
120	89	89	89	59	32	0	0	0
180	91	83	88	75	100	0	0	0
240	100	89	94	82	97	6	6	22

SUMMARY

The two ASTM methods available for evaluating the false set potential of portland cements are based on the loss in penetration within a few minutes after mixing.

The mortar method provides a fixed water-cement ratio, 30 per cent for cement types I and II and 32 per cent for type III. The penetration after remixing aids in distinguishing between quick and false set. The mortar method is recommended for research studies.

The paste method uses a specified initial penetration to establish the water requirement. The latest revision of this method incorporates machine mixing to reduce the human-factor influence on the water requirement. Optional limits for the paste method have been accepted by ASTM for the portland-cement specifications.

Since the behavior of many cements in these tests is seriously affected by the exposure received before testing, the use of plastic or metal containers for samples is recommended.

The presence of false set in a cement is no disadvantage for many applications. It is recommended that the purchaser should not specify the optional false set limits in the cement specifications unless false set will be detrimental in the use of the cement.

DISCUSSION

MR. W. C. HANSEN[1] (*by letter*). —The provisions in the specification used by the Hydro-Electric Power Commission of Ontario that the cement shall not show false set at 50 F recognizes the fact that the rates of reaction of the calcium aluminates may be a function of temperature. I reported[2] on a cement that prior to aeration reacted with calcium sulfate at a rate at which no false set developed although the cement contained some amount of calcium sulfate hemihydrate. Aeration affected the calcium aluminates in a way that reduced their rates of reaction with calcium sulfate sufficiently to permit the hemihydrate to cause the crystallization of gypsum and produce false set. It seems likely that the effect of a reduction in the temperature at which the unaerated cement was tested would have been similar to the effect of aeration. It would seem from this that users of cement should not specify tests at low temperatures unless the cement is to be used at such temperatures.

MR. J. L. GILLILAND (*author's closure by letter*).—Mr. Hansen's comments on the effect of temperature on the false set test are appreciated. Many factors enter into the SO_3 availability and requirement of the paste. The net effect of these factors establishes the false set characteristics of a given cement for a given set of conditions.

In order to clear up any misunderstanding, it should be noted that the "definitions" and "interpretation" given in this paper are revisions to ASTM Methods C 359 and C 451. These revisions have been accepted by the Working Committee on False Set, and will be presented to ASTM Committee C-1 on Cement for approval in December, 1963.

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[1] Valparaiso, Ind.

[2] W. C. Hansen, "Aeration Cause of False Set in Portland Cement," *Proceedings*, Am. Soc. Testing Mats., Vol. 58, 1958, p. 1044.

SIGNIFICANT TIME-OF-SET STUDIES AND THEIR POSSIBLE RELATION TO CURRENT TIME-OF-SET PROBLEMS*

BY E. E. BERGER[1]

SYNOPSIS

The methods of test for setting time by the Gillmore and Vicat needles have been in general use since the 1880's. Limitations of both methods of test have been recognized, and there have been differences of opinion concerning the relative merits of the two test methods for more than half a century.

Since 1928 the Working Committee on Time of Set, of ASTM Committee C-1 on Cement, has been attempting to improve these two methods of test, and other methods of test were investigated in the hope that a generally acceptable test method could be developed.

A brief review of the limitations in the test methods and a summary of the studies that have been made is presented.

Some reasons are given as to why the available data have not been considered conclusive enough for general acceptance, and some avenues for a new approach to the problem are suggested.

In 1818 Vicat (**1**)[2] described a method to determine the point beyond which the form of cement paste could not be altered without fracture. For this test a flat point was filed on a darning needle 0.12 cm in diameter, and the other end was stuck in a lead ball weighing 300 g. The Gillmore test, designed to measure the same end point, was described in the ASCE recommendations of 1885. Both methods of test were in extensive use in the 1880's (**2**).

The difference of opinion as to which test was the more suitable must have started about this time also as we find the Gillmore test limits given in the 1901 specification of the Board of Engineers while the first ASTM specification of 1904 refers to the Vicat test.

There is general agreement that the setting of any cementitious material is one of its essential properties. However, when the word "time" is added, the question of degree of consolidation arises, together with the problems of how to identify and measure a certain degree of consolidation.

It may well be expected that, in a paper such as this, some definite solutions to these problems would be presented; however, it must be stated that such solutions are not yet available. All that can be done is to present a review of the time of set studies over the past years to aid in planning a program for future studies. Some suggestions later in the paper may be worthy of consideration when this future planning is undertaken.

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* Presented at the Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

[1] Kosmos Portland Cement Co., Kosmosdale, Ky.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

Except for two slight changes in the Vicat test, the two methods of test for setting time have remained essentially unchanged since they were developed some 80 yr ago. This indicates one of three view points: (1) the methods of test are quite satisfactory in their present form; (2) there has been no interest in or study of the setting time test; or (3) the significance of the test is not clearly defined.

This paper gives consideration to each of these view points as they relate to the setting time problem.

CRITICISM OF TEST METHODS

The following quotation from Meade's book (3) is a good indication of the feeling that existed at that time and perhaps to some extent today:

> "While the Vicat needle unquestionably is a much more scientific instrument and should be used where great nicety is required in making the test, as in disputes, etc.; still for ordinary inspection work, where all that is needed is the assurance that the cement will not set before it is laid in position in the job, and that after it is placed it will harden in a reasonable time, the simpler and less expensive Gillmore will answer the purpose just as well as the more expensive Vicat apparatus. The Gillmore needles are the ones usually used by both the manufacturers and engineers . . . and most of those called upon to make the test are familiar with the terms initial and final set as defined by these needles, so that it does not seem to have been a very wise plan on the part of those formulating the rules to recommend that the tests be made with the Vicat needle. Setting time is influenced by so many things besides those over which the Vicat has control, that the personal equation is as much an element in a determination made with this apparatus as with those made by the Gillmore needles."

It may be of interest to remember that when the above statement was made, the Corps of Engineers was using the Gillmore test while the Vicat test was specified by the ASTM.

Other quotations also show that there had not been complete satisfaction with either of the setting-time test methods.

The most poignant comment may have been made by Cooper (4) who, in discussing the Vicat test, stated in part as follows: "The question of setting time is somewhat vexed, and I think more disputes arise between the user, the engineer and the manufacturer on questions of setting time than anything else . . . a cement which took two hours to set at the works takes two days or two minutes on the job. . . . This is a point on which very valuable research might be carried out."

The 1928 report of Committee C-1 on Cement includes the following statement: "Tests for normal consistency, fineness, and time of setting show a lamentable lack of concordance." By 1933 some improvement is indicated in the following statement by P. H. Bates (5): "Our present time-of-set measurements seem satisfactory if one will neglect the much mooted question of 'grab set' and the eternal question of what constitutes 'appreciable' indentation." Bates states further that the old squabble of Vicat versus Gillmore has mellowed since we have two needles and both sides won. However, the inference seems to be that we have two needles but no solution.

The 1936 report of the Working Committee on Time of Set included a statement on a survey made to determine how the time of set tests were meeting with the approval of those most concerned with the testing and use of portland cement:

"Although a considerable majority felt that the standard methods were satisfactory in every way, there has been persistent evidence to the contrary from various sources. To say the least, the strong difference of opinion regarding the relative dependability of the alternate methods provided does not reflect favorably on either method and in the opinion of many presents an undesirable situation."

A letter from a state highway engineer in 1940 states that we are unable to secure checks with most laboratories where the Gillmore was used but we have no difficulty with the laboratories in checking the Vicat method.

Blount (6) states that, "The Vicat needle is the only apparatus of any value in determining setting time"; but he states further that, "There is no quantitative relationship between the setting time of neat paste and its setting time in concrete." He concludes that, "Much earnest and thoughtful work must be done before we can arrive at anything which can be accepted as proven."

Schutz (7) in 1959 states that laboratory setting-time tests provide the engineer with little information as to when cement will stiffen in the field, and he added that the definition of setting time is broad, uncertain, and arbitrary.

The current chairman of the Working Committee on Time of Set has received many comments both for and against both test methods; but he has two letters from outside the United States that emphasize the wide use of the ASTM test methods and therefore the careful study that should be made before making a recommendation on any test method. The extensive use of ASTM test methods almost prompts the author to describe the long-time dissatisfaction that has existed with the methods of test for setting time as critical.

One letter contains over three pages of comments on the lack of precision in the normal consistency and the Vicat test. The other letter, accompanied by a well-marked Gillmore pat, asked, "Which of these points should be identified as appreciable indentation?"

SUMMARY OF SETTING-TIME STUDIES

Recognizing the various criticisms and opinions on the setting-time test, Mr. Bates, Chairman of Committee C-1 on Cement, wrote a letter to members of this subcommittee on June 8, 1940, in which we find the following statement: "Efforts should be made to develop procedures which would overcome the need for specifying appreciable indentation—an indentation which depends much on the whimsy of the operator and varies much between producer and consumer operators." In the same directive he suggested that the tests might be made on mortars and that the Vicat apparatus, rather than the Gillmore, suggests itself for use in these tests. The directive was repeated in a letter from Mr. Bates in 1943.

In view of the general dissatisfaction that has existed with the time-of-set tests, one would expect that a great deal of work would have been done to correct the situation. Actually more work has been done on the setting-time problem than is generally realized.

REVIEW OF TEST PROGRAMS

The lack of uniformity in cement testing was recognized early in this century. In 1912 a Joint Conference on Uniform Method of Test for Cement was established consisting of three representatives each from the ASTM, the American Society for Civil Engineers, and the Governmental departments.

First a questionnaire was distributed but since no general agreement was apparent a comprehensive series of tests was set up involving practically all laboratories having anything to do with cement testing. These included over 200

Government, state, municipal, college, private, and cement producer laboratories.

At the end of the first series of tests, the ASTM and the American Society for Civil Engineers gave eight reasons for favoring the Vicat test method while the Governmental group gave four reasons for favoring the Gillmore test together with reasons for nonagreement with some of the conclusions of the other two groups.

Eventually two additional series of cooperative tests were conducted. The final conclusion from the summary of all data was that the Gillmore test was 80 per cent as accurate as the Vicat test. But after a study of all these data and after nine sessions of the Joint Conference group, the differences among the three groups were not resolved. All the data and the minutes of the eight conference sessions were submitted to Committee C-1 on Cement, and as a result both the Vicat and the Gillmore methods of test were included in the 1917 ASTM specifications for cement.

The first cooperative series of tests on setting time sponsored by Committee C-1 appear to be those included with the other physical tests in 1928. In these tests neat fluid pastes were included along with cement pastes mixed to a normal consistency. Tests were run also with a 100-g needle as well as the 300-g needle. This probably was the most extensive series of tests ever conducted in Committee C-1 on Cement as 52 laboratories and 33 cements were involved in this series.

The deplorable lack of coordination reported in this series of tests demonstrated the need for a special laboratory as a recognized authority; thus, the movement to establish what is now the Cement and Concrete Reference Laboratory was started.

In 1934 the Time of Set Committee under the chairmanship of Roy N. Young considered a mortar test. The incentive for this work was the results obtained by Gause (8) at the National Bureau of Standards in his work with high-early-strength cements. Mr. Young describes his method of test and the results in his report to Committee C-1 in July 1936. This series of tests included the Vicat test, the Gillmore test, and a mortar test using mortar prepared in accordance with ASTM Method C 109.[3] Seven laboratories participated in these tests using six different cements. The committee after some study decided that a consistency test was essential because of the wide difference obtained with constant proportions of cement, sand, and water. A penetration test was used for this purpose.

Except that a C 109 mortar was used instead of a 1:1 mortar, the test was quite similar to C 229[4] which is published and need not be reported here. In fact, the results obtained with the mortar test in 1936 furnished the main incentive for the more extensive mortar studies begun in 1944.

In the report of the 1936 series of tests the divergence among laboratories including all cements and tests gave a mean variation of 14.5 per cent for the Gillmore test, 12.9 per cent for the Vicat test, and 7.8 per cent for the mortar test. Because of this increased uniformity of the mortar test, the report states that "its advantages lends encouragement toward the development of a test of this nature."

The Working Committee on Time of Set hoped to avoid the use of a special mortar for setting time. Since the strength committee was considering "baby concretes" at that time, further work on setting time of mortars was postponed pending a decision regarding the type of mortar to be used for the strength test. The setting time committee concerned itself with the problem of false set for which it was responsible until 1953; thus, their valuable work on the mortar test probably received less attention than the results warranted.

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[3] Method of Test for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. Cube Specimens) (C 109), 1961 Book of ASTM Standards, Part 4.

[4] Method of Test for Setting Time of Hydraulic Cement in Mortar (C 229), 1949 Book of ASTM Standards, Part 3.

Test Series, 1940:

From 1940 to 1962, the Working Committee on Time of Set conducted seven cooperative series of tests and a great deal of exploratory work was conducted together with a study of the problem of false set between 1947 and 1948.

These cooperative series will be summarized to avoid duplication of certain results in future studies of this setting problem.

In the first series of tests, reported to C-1 in July 1941, types I, II, and III cements were included together with an air-entraining cement and a masonry cement. Regular Vicat initial (35 mm penetration) and Gillmore initial tests were run together with fluid pastes and fluid 1:1 banding sand mortars. The fluid consistencies were mixed in a bowl with a domestic type mixer, and the 10-mm penetration of a 2-mm diameter needle was taken as the time of set. The average deviation among all the laboratories and all the portland cements used showed the mean variation on the Gillmore tests to be 14.0 per cent, the Vicat tests to be 12.7 per cent, while the neat fluid paste and the banding sand mortar both showed a mean variation of 5.2 per cent. The masonry cement showed 21 per cent mean variation for the Vicat test and 13 per cent for the Gillmore test; however, this variation was reduced to 6.1 and 3.8 per cent with the fluid mixes. In this series of tests, the small slump cone that was used for measuring consistency proved to be unsatisfactory. Furthermore the high speed of the mixer whipped too much air into the fluid mixes and the setting time seemed too slow for practical limits. Consequently, this type of test was abandoned even though uniformity was improved, and the water-cement ratio used was similar to that required in concrete.

The reader today may question the use of per cent mean variation, which is the mean of the absolute deviations from the average divided by the average result, rather than the coefficient of variation in the evaluation of data. At the time when most of these data were obtained they were not subjected to the detailed statistical analysis that is being used today. Since the results available were reported on the basis of per cent mean variation, this procedure was continued to give a direct comparison in evaluating the results of the various series of cooperative tests.

In this paper, both the per cent mean variation and the coefficient of variation are calculated for some of the results reported in 1947 to show that the evaluations were similar. The coefficient of variation was used in evaluating all subsequent data.

Before proceeding further with cooperative tests, the Working Committee on Time of Set decided that any end point chosen should have some definite significance. Standard liquid limit tests following Method D 423[5] were tried. Experiments were also conducted using plastic limit tests with "pie sections" taken from flow-table specimens; slump tests; early compression tests; and time penetration tests on both mortar and paste. One laboratory correlated their tests with slump tests on ready-mix concrete using the same cement. All tests indicated a break in the curve at some particular time, and when the same cement was used these times were related. It was only after analyzing these data from the various laboratories that the 10-mm penetration of a 300-g needle was adopted for the setting time of the 1:1 mortar.

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[5] Method of Test for Liquid Limit of Soils (D 423), 1961 Book of ASTM Standards, Part 4.

As a result of these exploratory tests the following definition for time of set was submitted to Committee C-1 in 1943: "It may be said that a mortar has set when it has attained such a degree of induration that its form cannot be altered without fracture, that is, when it has entirely lost its plasticity." This is an exact copy of the definition given by General Gillmore (9) in his book published in 1874, and it is quite similar to Vicat's definition of 1818 (1).

Test Series, 1944:

In a second series of cooperative tests conducted in 1944, types I, II, III, and I(*a*) cements were used in 14 different tests. This series of tests included: (1) the standard Vicat and Gillmore tests; (2) C 109 mortar test with consistency determined by the flow table; (3) three tests each with graded sand, banding sand, and ground sand as follows: (*a*) constant sand-cement mix with water varied to obtain a definite penetration with a 400-g, $\frac{11}{16}$-in. needle, (*b*) a mix with cement-water ratio constant and sand varied to produce a specified consistency, and (*c*) a constant mix; (4) one mix with varied amounts of water using standard sand; and (5) one special mix with a flint sand. Both a 1-mm and a 2-mm needle were used to measure setting time but the 1-mm needle was not recommended for future tests because of sand grain interference and the tendency for the needle to bend during use.

The report, submitted to Committie C-1 in 1946, shows that the average deviation for all laboratories and all cements gave a mean variation of 14.9 per cent for the Gillmore initial tests, 11.4 per cent for the Vicat tests (still a 35-mm penetration end point), 9.8 per cent for the C 109 mortar, 4.8 per cent for the 1:1 graded sand mortar, 6.1 per cent for the banding sand mortar, and 4.6 per cent for the ground-sand mortar. All the above mortar results were obtained on mixes in which the water was varied to obtain a certain consistency. The results, with a constant water-cement ratio and sand varied, were slightly better but not enough to justify the increased time and expense in testing.

Test Series, 1947:

While the results obtained in 1944 were quite encouraging, some of the laboratories were unable to complete the tests because of the stress of other problems during this critical period. Consequently, it was decided that a second series of tests should be conducted with a larger group of laboratories to check the findings on the most encouraging mixes. Seven laboratories and four cements were included in this series of tests conducted in 1947. In summarizing the data from these tests it was noted that the average deviation for the four cements gave a mean variation of 6.8 for the 1:1 graded sand mortar, 6.0 per cent for the 1:2.75 mortar (consistency checked by penetration method), 6.8 per cent for the ground sand mortar, and 5.6 per cent for a 50-50 mixture of ground sand and graded sand. This series of tests was preceded by preliminary tests on the effect of different sand combinations.

In the above series of tests the individual laboratories checked different rounds quite closely. A special series of tests with 1:1 mortar was then added in an attempt to resolve the differences between laboratories and to compare the mixing procedure with method C 185.[6]

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[6] Method of Test for Air Content of Hydraulic Cement Mortar (C 185), 1961 Book of ASTM Standards, Part 4.

The original mixing procedure gave the better results, the mean variation being 3.9 per cent for tap water and 2.6 per cent for distilled water. The coefficient of variation was 4.8 per cent and 3.1 per cent, respectively. Some laboratories obtained practically the same results with either tap water or distilled water, while in other cases there was a significant difference in results.

With the results of the 1947 series of tests, the Working Committee on Time of Set recommended the 1:1 mortar test to Committee C-1. The test was approved as a tentative method (C 229).

Favorable comments were received regarding the results obtained with the 1:1 mortar test; however, it was considered to be rather expensive and time-consuming for the large number of routine setting-time tests required. Consequently, the working committee was requested to develop an alternate method of test for general work.

Since previous studies had shown that more concordant results were obtained in the Vicat test, the program for the next series of tests was concentrated on modifications of the Vicat test. Nevertheless, Gillmore initial tests and 1:1 mortar tests were included also.

Test Series, 1949–1957:

One series of tests was made in 1949 involving four cements and eight laboratories. Then to check the more promising methods, a second series of tests was conducted in 1950 in which seven laboratories cooperated in the testing of four types of cement. Wetter mixes, different sized needles, and different type molds were tried in these two series of tests. Time-penetration tests were included also to find the end point giving the most concordant results. As a result of this work the 25-mm penetration is now specified as the Vicat set rather than the 5-mm from the bottom penetration that was considered the point of initial set previous to this time.

The average for these two series of tests show a mean variation of 12.8 per cent for the Gillmore initial test, 8.5 per cent for the Vicat initial, and 6.7 per cent for the 25-mm penetration of the Vicat needle. The other modifications of the Vicat test gave less concordant results than the one adopted.

Complete data on these two series of tests were presented to Committee C-1 in March 1951. The report also included a letter to the Sponsoring Committee giving comparative results obtained with the Gillmore, Vicat, and 1:1 mortar test to guide them in setting limits for the latter test.

After careful consideration of the data obtained, the Time of Set Committee decided to submit three recommendations: (1) Adopt Method C 229 as standard, (2) use the Vicat test as an alternate method with a minimum setting time of 45 min and a maximum setting time of 7 hr, and (3) retain the Gillmore method temporarily but delete any reference to it in the standard specifications. Later discussion indicated that such a complete change at one time was not advisable and these recommendations were never submitted formally to Committee C-1. A revised recommendation was submitted in March 1951 which resulted in the following action by C-1: (1) a tentative revision of Method C 229, (2) separation of the Vicat and Gillmore test methods, and (3) the footnote in the present Specification C 150[7] stating that in case of dispute the Vicat test results shall govern.

This change in recommendation of the Working Committee on Time of Set and the separation of the two test methods caused considerable confusion, and a great deal of correspondence was required before it was clarified. Because of this confusion, or for some other reason, the maximum limit for setting time by the Vicat needle has been omitted from Specification C 150 although it is included in Specification C 205.[8]

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[7] **Specification for Portland Cement (C 150), 1961 Book of ASTM Standards, Part 4.**

This maximum limit was suggested as a safety measure to insure normal hardening time and lack of contamination. The omission of this limit may be one reason why some people prefer the Gillmore method of test since this safety measure is provided in the Gillmore final-set test.

Since the main criticism of the 1:1 mortar test was the time and expense involved, consideration again was given to a C 109 mortar test which had given more concordant results than the neat paste tests although not as good as the 1:1 mortar test. The C 109 mortar had shown two disadvantages: (1) bleeding occurred in this mortar and (2) the wet mixes required to obtain 110 per cent flow with higher air contents gave slow setting time results. It was thought that the latter difficulty could be eliminated by using a penetration test rather than the flow table for measuring consistency. The Working Committee on Strength considered this, and penetration tests were included in the 1953 series of tests. However, it was reported that the manner in which they were used did not permit any conclusions. This matter has not been pursued further but it is worthy of consideration.

ASTM now had three methods for setting time, a situation obviously undesirable. Since the 1:1 mortar test was being used only occasionally for research work, it was discontinued in 1955 at the suggestion of the working committee.

This action restored the previous situation of two available methods of test and the existence of some difference of opinion as to which test was the better of the two.

In all cooperative tests with which the author is familiar, the Vicat test has proven to give more reproducible results than the Gillmore test when testing portland cements. However, a number of deficiencies in the Vicat test method are evident: nonuniform release of the needle, friction of the needle especially when it was not absolutely clean, bending of the 1-mm needle, and greater expense of the test. Therefore, in 1957, a task group within the Time of Set Committee ran tests with a penetrometer that had an automatic release mechanism. The results appeared encouraging; nevertheless, since all previous data apparently had failed to produce any increased acceptance of the Vicat method of test, the Working Committee on Time of Set could see no purpose in continuing this study.

The current study of the Vicat apparatus by the Working Committee on Bleeding and Plasticity may result in some improvements that will be helpful in the setting-time test.

Current Test Series:

Once more serious consideration was given to ways in which the Gillmore method of test might be improved. A ring around and slightly above the needle tip, similar to that used in England for the final set of the Vicat needle, was tested carefully by the Time of Set Committee, but no definite improvement was noted.

A series of cooperative tests involving eight laboratories was conducted in 1961 in which different modifications of the Gillmore test were tried. These tests included a longer mixing time, the use of $\frac{1}{2}$- by 3-in. rings along with the Gillmore pat, and an attempt to specify a more definite end point.

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[8] Specification for Portland Blast-Furnace Slag Cement (C 205), 1962 Supplement to Book of ASTM Standards, Part 4.

Here again, the Vicat tests gave more concordant results than any of the Gillmore tests and the modified end point only increased the confusion.

Unfortunately, one cement used in the 1961 test series developed some false setting tendencies after being prepared for distribution. There was some thought that this may have influenced the results. Since these data were to be related to certain current questions before the Time of Set Committee, this series of tests was to be repeated in 1962 with the same eight laboratories.

This last series of tests was delayed so that complete results are available from only six of the eight laboratories; therefore, no definite conclusions can be presented at this writing. The available data indicate that it will still be inadequate to answer some of the questions involved in the setting-time problem. However, the differences resulting from aging of the cement and the marked differences in results obtained by a slight change in the manipulation of the test specimen, even in the same laboratory, indicate that a careful study of these data will prove to be of value in planning the future studies of the Working Committee.

Time of set tests were included in a study of mechanical mixing reported by the Coordinating Committee in 1959. Eleven laboratories and nine types of cement were included in the summary of results. In this series the average coefficient of variation for the Gillmore and Vicat tests were 17.5 and 11.7 per cent, respectively. It was noted, however, that on the masonry-cement tests the coefficient of variation for the two methods of test was about equal.

MECHANICAL DEVICES

To make this report more complete some mention should be made of one other approach to this problem, namely, mechanical devices. Some of these are simply listed here so that they may be investigated further if this should prove to be fruitful.

The first mechanical device to be used was a loaded roller with fins pulled by clock work. This device was developed by Professor Goodman in 1887. Another device was an automatic penetration apparatus designed by W. S. Hodge. H. S. Meisner (10) of the Bureau of Reclamation developed an improvement of Hodge's device in 1950. An electronic device was designed by Tobio (11). Satava (12) describes an apparatus for automatically recording the weight required for a certain penetration, and Callega (13) gives extensive data obtained with an electrical resistance method.

The machines apparently performed satisfactorily from a mechanical standpoint, but because of certain limitations little was accomplished in solving the problem of time-of-set. No machine, whether it be a Gillmore needle or an electronic computer, can give a logical answer unless purposeful programming is fed into it; therefore, the solution to the problem can be found only in the minds of men, not in machines.

IMPORTANCE OF SETTING-TIME TEST

After exploring the differences of opinions, dissatisfaction, and lack of progress on the setting-time problem for over fifty years, it is obvious a reassessment of the importance of the problem should be made, and consideration should be given to the direction of further studies.

The setting of cements is its primary function and therefore the most important property of this material. The methods of test for setting time attempt to evaluate this important property on the basis of time. The difficulty is that the time obtained in the test is unrelated to concrete performance in the field.

Cooper (4) called this to our attention, and it has been confirmed again and again. Furthermore, since the time of set has not been definitely defined, it is possible that time does not evaluate all the cement-setting characteristics that are considered in connection with the test results.

If a sample of cement paste is not retarded adequately, an experienced technician will know it before he uses any needle. However, if the paste is still soft at the end of the day, then the cement is not normal. Generally, a needle is used, and a certain time is decided on.

The time depends upon characteristics of the cement but according to Williams (14), it depends also on the ambient conditions in the laboratory and storage cabinet. Mixing time and rate, troweling, exposure time out of the storage cabinet, and the operators' opinion as to what constitutes appreciable indentation are other factors that will affect the time selected. Consequently, different laboratories are not expected to check each other. The Vicat test method does a little better than the Gillmore in that less of the sample is exposed and the appreciable indentation limit is eliminated. However, there are other possible variations in the Vicat test method. Neither test gives an adequate indication of what can be expected in concrete.

Millions of barrels of normal setting cement have been produced where the Gillmore or Vicat test has been used to control the setting qualities of the cement. Moreover, technicians in the cement laboratory may detect certain variations in the manufacturing process by noting differences in the setting-time test. However, to apply such tests with all their possible errors to specification time limits, or to attempt to use them in evaluating cement quality, where different laboratories are involved is quite a different matter.

In developing the 1:1 mortar test, the Working Committee on Time of Set had two fundamental purposes in mind: (1) to obtain an end point that complied with Gillmore's plastic limit definition and (2) to develop eventually a method of test that would give a good indication concrete performance. As a first step it was thought that close agreement among laboratories in the setting-time test was essential if any correlation with concrete performance was ever to be attained.

The committee was rather successful in obtaining concordant results, and the report from a state highway engineer indicated that the 1:1 mortar test could be correlated with concrete setting time better than either the Vicat or Gillmore test. Whether this correlation is adequate, especially with the increased use of processing additions, can be determined only by further study.

CURRENT CONCEPTS

Definitions:

Presently, The Working Committee on Time of Set has been requested to recommend a definition of setting time. As noted earlier in this paper, Gillmore's definition was suggested to Committee C-1 in 1943, but little consideration was given to it for the following reasons: if the setting-time test is broad, uncertain, and arbitrary, as Shutz (7) indicates, then correlation of this with another indefinite term such as plastic limit can result only in a weak foundation from which to build a test method.

A similar problem was encountered in arriving at a definition for the setting time of concrete. Scripture (15) summarizes a rather extensive study of this problem and concludes in part as follows: "With respect to a definition of setting time of concrete, it is evident that this term means something different to each individual, depending on the specific application for which it is intended. . . .

Any definition therefore, awaits establishment of a suitable test method."

The immediate response may be that no such difference in purpose could exist in testing a more homogeneous system such as cement paste. However, since the relationship between the setting-time test and concrete performance is not established, the real purpose for this test is not clearly defined. Neither is there a clear indication of the significance of the end points that have been established. Consequently it is quite possible that the test for setting time could have a different significance for different people.

Our terminology may even be reevaluated. For initial set there always has been some idea of plastic limit in mind, but this point has not been definitely defined. The significance of the final-set test is even less definite. The only thing we know for sure is that it is not final set either in the case of a neat specimen or a concrete specimen.

Therefore, it could be that this then can be part of our difficulty, our definitions are not adequate. The first step, then, is to analyze the problem so that the purpose of the test may be well defined.

Recommended Test Method:

The Working Committe on Time of Set is also trying to determine the best method of test. It is not just a matter of preference.

On the basis of the large amount of data available, the Time of Set Committee has suggested, with limited success, that the status of the Gillmore test be reduced and emphasis given to the Vicat or a mortar test. This means that the methods of test or the evaluation of results, or both, simply failed to yield the information desired. Or it could mean that, subconsciously at least, we agree generally with the writers cited in this paper who state that the setting time recorded (from either method of test) has little significance in relation to concrete performance; therefore, it is not justifiable to exert the time and effort necessary to obtain precise measurements of this time. Consequently, it seems quite obvious that further tests following the programs of our previous studies will not be of much value in resolving the problem.

If the above reasoning appears to be facetious, cynical—or even critical—the reader may be assured that no such thoughts are intended. The long-standing problem has been attacked vigorously by one approach and without success, it is now time to try a new approach.

It is proposed that a task group be chosen to analyze this problem and decide what setting characteristics are to be determined; then let these findings be submitted for suggestions and criticism. After the problem has been rather definitely defined, then the Committee on Time of Set should be in position to consider test methods. In the beginning, the needles should be kept out of the picture; otherwise the controversy may start all over again.

Should the starting point be with the definition or the test method?

In Method of Test C 403,[9] the following statement is noted: "Since hardening of concrete is a gradual process, definition of time of setting must necessarily be arbitrary. Times of initial and final setting of concrete are determined by this method on the basis of a rate of hardening test made by means of Procter penetration resistance needles on mortar sieved from the concrete mixture." (Note that this is really a mortar test.)

While this end point is considered an arbitrary limit, it is known from Scripture's paper that considerable effort was expended to make this limit as practical as possible. It is known also that the decision to base the definition upon a method of test was made only after a serious attempt to arrive at a suitable definition proved unsuccessful. Although the arbitrary end point established may have little theoretical significance, it can be used and understood.

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[9] Method of Test for Time Setting of Concrete Mixtures by Penetration Resistance (C 403), 1962 Supplement to Book of ASTM Standards, Part 4.

Such a test method for concrete obviously is not practical for use in the acceptance testing of cement, but, since such a test is now available, this may be the logical basis from which to start in developing a test method for cement.

The test method for cement would then be designed to have a method that gives a definite correlation with the results obtained in the C 403 test method, and to have a close concordance of results among laboratories using the test method so that the results in the cement test have some real significance.

Such a relationship has been one of the needs of the time-of-set test on cement for some time, and, with the more extensive use and need for evaluation of processing additions, such a relationship may be even more essential now than it has been in the past.

Whether an acceptable test for cement can be obtained with the use of a neat paste is questionable. Past experience has indicated that it cannot. Still the performance of mortar or concrete depends primarily upon the reactions within the cement paste itself. In 1941 Powers (16) stated in part as follows:

> "Indeed, the only factors that need to be considered in this connection when comparing pastes and concretes are the usual differences in water cement ratio and temperature. If these differences were eliminated, taking precautions to prevent the neat paste from acquiring an unduly high temperature, there is little reason to doubt that the rate of loss of plasticity of the concrete could be predicted from the rate of loss of plasticity of the paste alone."

It would appear that there must be a measurable relationship between paste and concrete. Perhaps the fluid pastes tried in 1928 and 1941 could yield the desired information if proper measurements could be developed. However, since more processing additions are being used today and more special cements are being considered than in 1941, the evaluation or acceptance of cements by the use of any neat paste test must be weighed very carefully.

On the other hand, a rich mortar test has shown more concordant test results, and there are indications that these results can be correlated with concrete performance more definitely than any neat paste tests. Therefore, a mortar test, with probably a higher water-cement ratio than that used in the 1:1 mortar test, may provide the desired results. Only further study can determine the solution.

Now, even if the time problem is resolved, the assurance of a satisfactory setting performance is not complete. There still remains the regulation of false set and optimum SO_3 content. Or possibly, if false set is not present and the optimum SO_3 content is attained, the measure of setting time may lose its importance as far as cement acceptance tests are concerned. This also is a problem for the hypothetical task force to consider.

SUMMARY

The early development of the present methods of test for setting time emphasizes the fact that the properties of the cementitious materials could have been quite different from those in use at the present time. Attempts, over the years, to adapt these tests to current problems and changes in cements has not been satisfactory to all concerned. Thus an analysis of our current problems

and a reassessment of our test methods is thought to be essential if a satisfactory test for setting is to be developed.

Acknowledgments:

Appreciation is expressed to the members of the Working Committee on Time of Set who have cooperated in the several attempts to resolve this problem, and to the many other members of Committee C-1 who have joined in cooperative tests and have submitted individual data and valuable suggestions to aid in the study of the setting-time problem.

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(9) Q. A. Gillmore, *Practical Treatise on Limes, Hydraulic Cements, and Mortars*, New York, N. Y. (1874).

(10) Bureau of Reclamation *Report No. CH-98* (1950).

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(14) G. M. Williams, "Errors in the Method of Determining the Time of Setting of Cement," *Proceedings*, Am. Soc. Testing Mats., Vol. 2 (1914).

(15) E. W. Scripture, Jr., "Setting Time," Symposium on Significance of Tests and Properties of Concrete and Concrete Aggregate, *ASTM STP No. 169*, Am. Soc. Testing Mats., p. 53 (1955).

(16) T. C. Powers, personal communication to Time of Set Committee (1941).

DISCUSSION

MR. M. A. SWAYZE[1] (*presented in written form*).—Mr. Berger's highly interesting paper on time of set studies and their relation to present set problems has been carefully studied by the writer, especially as to the relative merits of the Gillmore and Vicat tests and their comparative values in indicating setting time of concrete. In looking back over the 51 years of my experience with cement testing for setting time and other properties, it appears that we in ASTM Committee C-1 on Cement have been too much interested in recent years in the improvement of the precision of gadgets, and too little in the original purposes of the tests and in the changes in cement properties that might make the gadgets or test methods obsolete. Testing, especially for setting time, would become more meaningful if we would first consider the primary purpose involved rather than the exact procedures that should be specified in the latest test methods.

The original purpose of setting-time tests undoubtedly was to prevent use of quick-setting cements, or use of others bordering on hydraulic limes whose set was exceedingly slow. For rejection of quick-setting cements, the use of low water-cement ratios in neat pastes was a wise choice, since the dense, stiff paste would set faster than the wetter mortars or concretes. Prior to 1917, when the SO_3 limits for cement were raised from 1.75 per cent to 2.00 per cent, a number of high C_3A cements were usually dangerously close to quick set if the SO_3 limits were carefully observed by the producer.

Improvement in the hardening rates of portland cement over the past 30 years has certainly obviated any further need for final set limits in either Specification C 150[2] or Specification C 175.[3] For those who wish some knowledge of early rates of strength gain, a 24-hr strength test for all types of portland cement would appear to be more appropriate. Hence this discussion is limited to initial sets only.

Coming to the principle on which all setting-time determinations should be based, no better definition has ever been written than the one by Gillmore, which Mr. Berger has quoted: "It may be said that a mortar has set when it has attained such a degree of induration that its form cannot be altered without fracture; that is, when it has entirely lost its plasticity." Mr. Berger says Vicat's definition was similar. In modern parlance, set has occurred when the mixing water in either paste, mortar, or concrete has ceased to be the continuous phase, and the hydration products plus the remaining unhydrated cement have taken over.

As I discovered in 1912, while making Gillmore tests for initial set, prior to the incidence of initial set under the Gillmore needle free water can be brought to the surface of a pat by any sort of pressure—either by the initial needle or by a finger. Once initial set has been attained no water will appear, no matter how heavy the pressure. Following this observation, my pats more closely resembled a Bertillon fingerprint record than they did the usual setting-time pats, due to so few marks from the Gillmore needle. For neat pastes, as for mortars or concretes, failure to find a water film on the pat surface on lifting the finger denotes incidence of initial set just as well as the Gillmore needle itself. In fact, there is usually only 5 to 10 min between that time and that when "no appreciable indentation" is found for normal setting cements. I have always believed that Gillmore had made this same observation on effect of pressure and designed his initial set needle accordingly. His choice of the odd $\frac{1}{12}$-in. fraction for the needle diameter lends some credence to this deduction.

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[1] Cement Consultant, Scarsdale, N. Y.

[2] Specification for Portland Cement (C 150), 1961 Book of ASTM Standards, Part 4.

[3] Specification for Air-Entraining Portland Cement (C 175), 1961 Book of ASTM Standards, Part 4.

In contrast to this simple correlation between presence of water as a continuous phase in cement pastes and the lack of initial set in the Gillmore test, the Vicat test with its arbitrary 25-mm penetration seems purely empirical in nature, and therefore might well be abandoned. The important point for initial set, regardless of how determined, is that prior to its incidence the surface can be reworked without damage; after it, only harm will be done by any finishing method. Members of Committee C-9 on Concrete and Concrete Aggregates interested in initial set of mortars and concrete might well consider the use of this simple but effective means of set determination for their material.

Concerning future activities of the Working Committee on Set, I believe that it is futile for it to attempt further improvements in agreement between laboratories on time of set of neat pastes by either Gillmore or Vicat method unless they insist on a closer range of laboratory temperature and humidities, and an improvement in uniformity of tests for normal consistency. Use of a mortar instead of the neat paste would obviously improve agreement between laboratories but would lessen the chance of detecting marginal quick set in cements. As long as concrete is made with different water-cement ratios, with additions or admixtures of accelerators, retarders, water-reducing agents, or combinations of these, and then subjected to the wide range of curing temperatures running from winter cold to summer heat, I see no hope of ever obtaining any good correlation between the setting time of cement as determined in the testing laboratory and the set of concrete in the field. I do urge, however, that the Working Committee take another good long look at the Gillmore initial set test in the light of this writer's comment earlier in this discussion, and then reevaluate the respective merits of the two means for determining initial setting times.

MR. P. K. MEHTA[4] (*by letter*).—I would like to take issue with the author's opening statement: "In 1818 Vicat described a method to determine the point beyond which the form of *cement paste* could not be altered without fracture." According to the literature, Joseph Aspdin (1824) is the commonly acknowledged inventor of portland cement. Some authors, for instance Michaelis, give credit to I. C. Johnson (1845) as the true pioneer of the portland cement industry. The manufactured cementitious materials prior to 1845, therefore, consisted actually of hydraulic limes. I am, therefore, doubtful that the Vicat procedure of 1818 was developed for cement pastes. In fact, the French reference quoted by Mr. Berger is entitled "Experimental Investigations on Construction Limes."

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[4] Graduate Research Engineer, College of Engineering, Department of Civil Engineering, Division of Structural Engineering and Structural Mechanics, University of California, Berkeley, Calif.

MR. E. E. BERGER (*author's reply by letter*).—The title of the paper refers to time of set studies and problems and there is considerable discussion on test methods. There is no indication regarding limitation on the type of cementitious materials.

The test methods may have been quite satisfactory when originally developed. Vicat was considering hydraulic limes and Gillmore was primarily interested in natural cements (hydraulic lime is a cement).

These early tests were mentioned to emphasize the above facts and the tests over the years were summarized to show, as stated in my paper, that attempts to adopt these test methods to current problems and changes in cement have not been satisfactory.

It is gratifying to learn that Mr. Mehta's careful study of the paper has produced some thinking that I had hoped would develop.

The dates on which portland cement was developed are well known to any reader who would be interested in the time-of-set problem so it seemed unnecessary to mention them again in the paper.

MESSRS. N. G. ZOLDNERS AND V. M. MALHOTRA[5] (*by letter*).—The author is to be commended for presenting an excellent review of the methods of test for setting time of cement. The writers do not intend to enter into the controversy regarding the methods for determining time of set, but wish to offer some additional data obtained during a cooperative study in Canada that may be of interest to the Working Committee on Time of Set.

The Subcommittee on Physical Requirements and Test Methods of the Committee on Hydraulic Cements, Canadian Standards Assn., is conducting a cooperative testing program as a means for testing laboratories across Canada to evaluate their testing procedures and techniques. The Construction Materials Section of the Canada Department of Mines and Technical Surveys is acting as a coordinator for this program.[6]

The first phase of the progam which has recently been completed consisted of the distribution over a period of 18 months of identical test samples, prepared from five different cement lots, to 27 participating laboratories. The first samples were distributed in October 1960.

The participants in the cement testing program were the laboratories of every cement plant, 20 across Canada, two laboratories of Government corporations, one Provincial Department of Highways laboratory, two commercial testing laboratories, one municipal laboratory, and one Federal Department of Public Works laboratory.

In order to provide each participating laboratory with as uniform a test sample as possible, each 5-sack cement lot was blended for 1 hr in a Silex-lined 4 by 8-ft drum of a Taylor ball mill and then dumped into a stainless steel tank. The entire lot was then divided by successive riffling operations into 32 individual samples each weighing about 13 to 15 lb. One of these samples was split further into 32 smaller portions by riffling, resulting in 75-g samples for chemical analyses.

Each participant received one large

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[5] Head, and Scientific Officer, respectively, Construction Materials Section, Mineral Processing Division, Department of Mines and Technical Surveys, Ottawa, Ont., Canada.

[6] N. G. Zoldners and V. M. Malhotra, "CSA Cement Testing Programme, Phase I," *Mines Branch Investigation Report IR-602*, Dec., 1962.

TABLE 1.—SUMMARY OF STATISTICAL ANALYSES OF TEST RESULTS.

Description of Test	Number of Tests	Unit	Maximum	Minimum	Average	Standard Deviation	Coefficient of Variation, per cent
Sample 1:							
Normal consistency:							
Water/cement	22	per cent	25.0	23.0	24.1	1.16	4.8
Rod penetration	15	mm	11.0	9.0	9.8	0.77	7.8
Vicat:							
Initial time of setting	21	hr:min	3:45	2:35	3:00	0:20	11.1
Final time of setting	19	hr:min	7:30	4:00	5:18	0:55	17.3
Gillmore:							
Initial time of setting	22	hr:min	4:00	1:25	2:52	0:40	23.2
Final time of setting	22	hr:min	7:30	2:45	5:10	1:04	20.6
Sample 2:							
Normal consistency:							
Water/cement	25	per cent	26.0	24.0	24.75	0.47	1.9
Rod penetration	20	mm	11.0	9.0	9.77	0.56	5.7
Vicat:							
Initial time of setting	25	hr:min	4:15	2:40	3:25	0:25	12.2
Final time of setting	25	hr:min	7:35	3:45	5:25	0:55	16.9
Gillmore:							
Initial time of setting	23	hr:min	4:00	2:15	3:15	0:25	12.8
Final time of setting	23	hr:min	6:40	4:00	5:10	0:40	12.9
Sample 3:							
Normal consistency:							
Water/cement	25	per cent	26.0	24.0	24.8	0.55	2.2
Rod penetration	21	mm	11.0	9.0	9.8	0.82	8.4
Vicat:							
Initial time of setting	25	hr:min	2:50	1:55	2:23	0:19	13.4
Final time of setting	25	hr:min	5:25	3:10	4:07	0:37	15.1
Gillmore:							
Initial time of setting	26	hr:min	3:30	2:00	2:32	0:23	15.0
Final time of setting	26	hr:min	7:00	3:20	4:23	0:84	19.1
Sample 4:							
Normal consistency:							
Water/cement	23	per cent	29.0	22.8	25.6	1.2	4.9
Rod penetration	19	mm	11.0	9.0	10.0	0.77	7.7
Vicat:							
Initial time of setting	24	hr:min	3:30	2:15	2:51	0:12	7.3
Final time of setting	23	hr:min	5:55	3:20	4:43	0:22	7.7
Gillmore:							
Initial time of setting	23	hr:min	5:20	2:05	2:59	0:42	23:3
Final time of setting	23	hr:min	7:00	2:40	4:57	0:55	18.5
Sample 5:							
Normal consistency:							
Water/cement	24	per cent	27.5	24.0	25.57	0.88	3.4
Rod penetration	19	mm	11.0	9.0	9.53	0.68	7.1
Vicat:							
Initial time of setting	23	hr:min	4:19	1:30	3:09	0:40	21.0
Final time of setting	23	hr:min	5:55	3:20	5:02	0:36	12.1
Gillmore:							
Initial time of setting	24	hr:min	5:10	2:10	3:18	0:42	21.3
Final time of setting	24	hr:min	7:05	3:10	5:18	0:49	15.5

sample for physical tests and a smaller one for chemical analysis.

In addition to chemical and mortar strength tests and fineness and soundness determinations, time-of-set tests were carried out. Both Vicat and Gillmore methods were used for the time-of-set studies, the actual testing procedures being those of the Canadian Standards Assn. code (A5-1951) for portland cements. These procedures are generally identical with those of the ASTM Methods C 187,[7] C 191,[8] and C 266.[9] However, the setting-time determination by Vicat needle differs from the corresponding ASTM procedure in the following respects:

The ASTM procedure as outlined in Method C 191 makes no mention of the initial and final setting times but merely states "Record the results of all penetration tests and by interpolation determine the time when a penetration of 25 mm is obtained. This is the setting time." Section 7.5.3.1 of the above mentioned Canadian Code states "The initial set shall be said to have occurred when the needle penetration of 25 mm is obtained in 30 sec after being released, and the final set when the needle does not sink visibly into the paste."

A statistical summary of the test results is given in the accompanying Table 1. The analysis shows that the coefficients of variation for the methods for determining setting time of cement are generally high and vary between 7.3 and 23.3 per cent, indicating that the methods in themselves need improvement. Further, the coefficients of variation show that the values for the Vicat method are lower than those for the Gillmore method, except for the Vicat "final set" value for sample 2. This appears to be in agreement with the author's experience that the Vicat test has proved to give more reproducible results than the Gillmore test when testing different cements.

The interlaboratory cement testing program is being continued and it is hoped that upon its completion it will yield some useful and interesting information.

MR. E. E. BERGER (*author's closure*).—The discussions of this paper are very much appreciated. In general, they emphasize some of the points made in the paper. Mr. Mehta reminds us that the test methods were not designed for portland cement, which emphasizes the need for a reappraisal of these tests. Mr. Swayze's comments in the first paragraph are especially pertinent and will bear repetition: "Testing, especially for setting time, would become more meaningful if we would first consider the primary purpose involved rather than the exact procedures that should be specified in the latest test methods." On the other hand, if our purpose is to measure time I question whether another long look at the Gillmore initial set test will provide the answer. This test and modifications of it have been studied by the Time of Set committee with little success, and the valuable data submitted by Messrs. Zoldners and Malhotra confirm the suggestion in the paper that neither the Vicat nor the Gillmore test seems adequate if time is considered to be important. It seems evident that these discussions enhance the suggestion in the paper that our purpose must be definitely defined before an adequate test method can be developed.

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[7] Method of Test for Normal Consistency of Hydraulic Cement (C 187), 1961 Book of ASTM Standards, Part 4.

[8] Method of Test for Time of Setting of Hydraulic Cement by Vicat Needle (C 191), 1961 Book of ASTM Standards, Part 4.

[9] Method of Test for Time of Setting of Hydraulic Cement by Gillmore Needles (C 266), 1961 Book of ASTM Standards, Part 4.

USE OF X-RAY EMISSION SPECTROSCOPY IN CHEMICAL ANALYSIS OF CEMENT, RAW MATERIALS, AND RAW MIX

By O. E. Brown[1]

Synopsis

Much interest has been shown in the application of X-ray emission spectroscopy, often incorrectly referred to as X-ray fluorescence, for the analysis of cement, raw materials, and raw mix. This paper details some of the inherent problems encountered at the Research Laboratory of the Marquette Cement Manufacturing Co. over the past five years and their solution.

Various factors pertaining to equipment operation, particle size, and matrix effects are discussed and evaluated. Typical calibration curves for various methods of sample preparation are presented and statistical accuracies and precisions are stated. Use of X-ray emission spectroscopy is suggested for cement plant process control functions where stipulated practical considerations exist.

The proposed method uses fine grinding and internal standard corrections for optimum accuracy and speed of analysis. The paper suggests a cooperative study by Committee C-1 on Cement to evaluate the acceptance of this technique as a tentative method of test for cement.

The general theories and fundamentals of X-ray emission spectroscopy are well documented in the literature.[2, 3]

A Philips Electronics (Norelco) standard laboratory X-ray apparatus was acquired in 1958 for the purpose of evaluating the application of X-ray emission spectroscopy for the analysis of cement, raw materials, and raw mix. The single-sample helium chamber was replaced with the three-position helium chamber in 1959 and the four-position vacuum chamber in 1960. The data presented herein were obtained with the vacuum chamber. The limitations of the two helium chambers will be discussed later in the paper.

Early work disclosed that there were three basic factors to be considered and resolved before X-ray emission spectroscopy could be accepted and used. Fortunately the program of study was such that the problems were immediately evident. Unfortunately, however, the problems were not quickly resolved.

Simple briqueting of samples of cement gave X-ray results that deviated significantly from the accepted gravimetric results, and the deviations were considerably greater than could be accounted for by counting statistics alone. This produced immediate evidence of the effect of particle size, because types I and III cements generally gave parallel but not identical calibration curves. The effects of matrix variations were not readily obvious.

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[1] Engineer of Tests, Marquette Cement Manufacturing Co., Chicago, Ill.

[2] L. S. Birks, *X-ray Spectrochemical Analysis*, Interscience Publishers Inc., New York, N. Y., 1959.

[3] H. A. Liebhafsky, H. G. Pfeiffer, E. H. Winslow, and P. D. Zemany, *X-ray Absorption and Emission in Analytical Chemistry*, John Wiley & Sons, Inc., New York, N. Y., 1960.

Attempts were made to grind the cement samples, using equipment available in the laboratory for such purpose. The results were encouraging although the desired accuracy was still not achieved.

The early work of Claisse[4] suggested that the sample be fused in borax as a means of eliminating particle size effects. This technique was adopted and modified for the analysis of cement. It was found necessary to correct the weight of sample used for the ignition loss of that sample. Although some improvement was achieved in the X-ray results, this technique also failed to provide accuracies comparable to wet gravimetric analysis, and the effects of matrix variations within the samples were suspect. The available literature suggested three methods of approach; mathematical correction, attenuation or sample dilution, and the use of internal standards. Because of the variety of samples to be analyzed it was believed that the development of mathematical formulas would be much too involved and that sample dilution would greatly increase time of X-ray analysis. A program of study was developed for the use of internal standards to apply proper correction.

It was not easy to select internal standards suitable to correct for matrix effects. Vanadium was chosen as the internal standard for the higher atomic number elements found in cements. An internal standard for the lower atomic number elements that would not be present in the cement samples was much more difficult to select. Chlorine was considered, but the use of fused pellets made this choice impossible due to volatilization. It was therefore felt that phosphorus would be used although it was recognized that the materials being analyzed would at times contain sufficient quantities of phosphorus to make the correction for matrix inaccurate.

The use of fused pellets was actually more of an art in their preparation than a science. Because of this and other undesirable features, efforts were again concentrated to acquire suitable grinding equipment that would eliminate the particle size effects. A Bleuler mill which was reportedly being used successfully

TABLE 1.—INSTRUMENT SETTINGS FOR PHILIPS STANDARD LABORATORY VACUUM X-RAY.

Element	Crystal	Theoretical 2θ Angle, deg	Pulse Height Analyzer Settings		Number of Counts
			Base Line	Channel Width	
Magnesium	ADP[a]	136.47	12.0	15.0	4 000
Aluminum	EDDT[b]	142.40	14.0	21.0	32 000
Silicon	EDDT	107.98	17.5	21.0	128 000
Phosphorus	EDDT	88.66	22.0	21.0	32 000
Sulfur	EDDT	75.16	27.0	21.0	64 000
Chlorine	EDDT	64.93	33.5	21.0	64 000
Potassium	EDDT	50.28	none	...	256 000
Calcium	EDDT	44.84	none	...	2 048 000
Vanadium	EDDT	33.02	none	...	512 000
Iron	EDDT	25.39	none	...	512 000

[a] ADP, ammonium dihydrogen phosphate.
[b] EDDT, ethylene diamine *d*-tartrate.

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[4] F. Claisse, *Norelco Reporter*, Vol. III, No. 1, 1957, p. 3.

to reduce powders to very small micron diameters was acquired and a reinvestigation of the use of pressed pellets was initiated.

Simple grinding of samples of cement provided improved accuracy over that where no grinding was performed, but the accuracy and precision was still not comparable to wet gravimetric analysis. The system of internal standards was restudied and since elevated temperature was no longer employed in preparation of pellets, vanadium and chlorine (in the form of sodium chloride) were interground with the samples of cement with much improved success. Accuracy and precision are comparable to wet gravimetric analysis under these conditions.

TABLE 2.—TABLE OF VARIATIONS FOR FIXED COUNT OPERATION STANDARD DEVIATION VERSUS NUMBER OF COUNTS.

Number of Counts	1-σ	2-σ	3-σ
400	5.00	10.00	15.00
800	3.54	7.08	10.62
1 000	3.16	6.32	9.48
1 600	2.50	5.00	7.50
2 000	2.24	4.48	6.72
3 200	1.77	3.54	5.31
4 000	1.58	3.16	4.94
6 400	1.25	2.50	3.75
8 000	1.12	2.24	3.36
12 800	0.88	1.76	2.64
16 000	0.79	1.58	2.37
25 600	0.63	1.26	1.89
32 000	0.56	1.12	1.68
51 200	0.44	0.88	1.32
64 000	0.40	0.80	1.20
102 400	0.31	0.62	0.93
128 000	0.28	0.56	0.84
204 800	0.22	0.44	0.66
256 000	0.20	0.40	0.60
512 000	0.14	0.28	0.42
1 024 000	0.10	0.20	0.30
2 048 000	0.07	0.14	0.21
4 096 000	0.05	0.10	0.15
6 144 000	0.04	0.08	0.13
8 288 000	0.03	0.07	0.10

The technique that has been developed for the analysis of cement is equally suited to the analysis of raw materials and raw mix. Practical considerations make it desirable to eliminate the fine grinding and internal standards when application studies indicate that these are not required. Similarly, if fine grinding without internal standards will provide the desired accuracy, much time can be saved in the over-all analysis by eliminating the use of the internal standards. Such application studies apply only to the use of X-ray emission spectroscopy by a single cement plant where the characteristics of the raw materials, raw mix, or cement do not vary significantly from day to day, month to month, or year to year. Where there is any question, the use of internal standards and fine grinding should be employed.

TEST APPARATUS

Instrument settings and analyzing crystals used for the determination of each element are listed in Table 1. The X-ray generator was operated at 45 kv and 40 ma. The pulse height analyzer settings shown in Table 1 are made by using a pure sample of silicon and adjusting the detector high voltage to provide maximum response at the indicated setting for silicon. Pulse height settings for the remaining elements are calculated from that of silicon. A flow proportional counter was used for all measurements and the X-ray tube contained a tungsten target.

Statistical accuracy was achieved through the use of a constant count technique. The number of counts required to achieve a 3-σ counting error acceptable for comparison with gravimetric analysis is listed in Table 1 for a pressed ground sample containing the internal standards. It should be noted that the number of counts required is based upon the concentration of the element present in accordance with the table of variations as standard deviation versus number of counts shown in Table 2.

The single-position helium chamber and the three-position helium chamber originally used would permit the introduction of only a single sample into the analyzing chamber at any given time. This did not provide sufficient accuracy and precision since changing gas conditions contributed to fluctuations beyond the limits acceptable for the analysis. Because counting statistics were selected on the basis of a 3-σ deviation of ±0.3 per cent of the amount of the element present, the subsequent shift of the calibration curve by an amount equal to ±0.3 per cent meant that actual accuracy was no better than ±0.6 per cent of the element present. The four-position vacuum chamber, which provides for all samples being in the vacuum chamber simultaneously, fulfilled the need to eliminate the shift of calibration curves. Other types of X-ray equipment are equally suited if the reference standard and unknown samples are in the chamber and subject to analysis without changing the conditions of the chamber. Equipment that does not provide for at least the reference standard and one unknown being in the chamber at the same time cannot provide this accuracy.

TABLE 3.—COMPARISON OF WET GRAVIMETRIC AND X-RAY ANALYSIS OF CEMENTS USING FUSED PELLETS.

Number of Cement Samples, 12

	Standard Deviation of Differences Between Results of X-Ray and Gravimetric Analysis			
	SiO_2	Al_2O_3	Fe_2O_3	CaO
Without internal standard correction	0.32	0.16	0.05	0.44
Internal standard correction applied	0.21	0.18	0.05	0.33
	Standard Deviation			
Wet gravimetric analysis	0.18	0.24	0.13	0.35

SAMPLE PREPARATION

Specimens for the two methods of preparation that have been studied, namely, the fused pellet and pressed pellet technique, are prepared in the following manner:

Fused Pellets:

Exactly 7.000 g of a cement sample, calculated on the loss free basis, and 8.500 g of lithium tetraborate are fused for 10 min at 2000 F in a platinum crucible. After the crucible has been in the furnace for 5 min it is removed and the melt is swirled. The crucible is then placed back into the furnace for an additional 5 min. The melt is then removed from the furnace and poured into a 1¼-in. split ring set on a chromium-plated nickel plate maintained at a temperature of 750 F. The chromium plating is required to prevent sticking and contamination. The ring containing the glass pellet is allowed to cool slowly by moving it to a piece of masonite that has been brought to the temperature of the hot plate. The glass pellet is removed from the ring after about 10 min and set aside to cool, placing a small beaker over the pellet to protect it from currents of air. After the pellet has cooled completely, it is ready for exposure to the X-rays and subsequent analysis.

When internal standards are required, 0.150 g of vanadium pentoxide (V_2O_5) and 0.700 g of sodium acid phosphate (Na_2HPO_4) are added to the sample and lithium tetraborate prior to fusing.

Streaks and striations in the pellet are evidence of inhomogeneity, and the pellet must be discarded or remelted and repoured. The same procedure should be followed with pellets that crack or break because of improper cooling and with pellets that are obviously faulty. Grinding and polishing of the glass pellets is not necessary as long as the surface of the pellet is flat and free from imperfections.

Pressed Pellets:

Exactly 2.0000 g of a cement sample and 0.1000 g of sodium lauryl sulfate (a grinding aid) are weighed into the Bleuler mill and ground for exactly 6 min. This grinding reduces the particle size of the cement to $-20\,\mu$ which has been found adequate for the required accuracy. After grinding, the sample is brushed from the mill, placed in a $1\frac{1}{4}$-in. mold, and cellulose powder is added on top of the ground sample to provide suitable backing material to assist in handling the pressed pellets. The pellet is pressed at 8000 psi.

When internal standards are required 0.0400 g of vanadium pentoxide (V_2O_5) and 0.0400 g of sodium chloride (NaCl) are added to the sample before it is ground in the Bleuler mill.

TABLE 4.—COMPARISON OF WET GRAVIMETRIC AND X-RAY ANALYSIS OF CEMENTS USING PRESSED POWDER PELLETS.

Number of Cement Samples, 10

	Standard Deviation of Differences Between Results of X-Ray and Gravimetric Analysis						
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O
Unground without internal standard correction	0.73	0.16	0.07	0.98	0.19	0.34	0.02
Ground without internal standard correction	0.46	0.18	0.08	0.58	0.13	0.13	0.02
Ground with internal standard correction	0.17	0.14	0.03	0.24	0.11	0.06	0.02
	Standard Deviation						
Wet gravimetric analysis	0.22	0.28	0.10	0.35	0.17	0.10	0.04

EXPERIMENTAL RESULTS

The samples chosen for preparation of calibration curves were selected from the National Bureau of Standards Special Reference Sample Program of 1958 and the Marquette Interplant Series so as to provide samples representing virtually all types of cement and those on which an appreciable number of laboratories had performed the gravimetric analyses. Due to a change in the samples, analyses were corrected on the basis of the ignition loss, determined at the time the samples were used.

Table 3 contains the calculated standard deviation of differences between results of X-ray and gravimetric analysis using fused pellets with and without internal standard correction applied for SiO_2, Al_2O_3, Fe_2O_3, and CaO. Included is the standard deviation of wet gravimetric analysis determined from the results of the previously mentioned series. Types IS and V cements, which were later found to exhibit greatest matrix effects, were not included. While the results obtained using internal standard correction show essentially the same standard deviation as that of the gravimetric analysis, the time required in predetermining the ignition loss, in applying the correction for the weight of sample to be used, in preparation of the glass pellet, and for X-ray analysis resulting from the dilution of the sample makes this technique less acceptable than the grinding technique using internal standards. Furthermore, the fact that phosphorus must be added as an internal standard when fused pellets are used makes this method less desirable. It should also be noted that the dilution effect is so great that the determination of magnesium on fused pellets is impractical.

TABLE 5.—REPRODUCIBILITY OF X-RAY ANALYSIS OF CEMENTS USING PRESSED POWDER PELLETS WITH INTERNAL STANDARD CORRECTION.

Trial	IP 88	IP 89
1	63.07	62.86
2	63.25	62.96
3	63.23	63.05
4	63.18	62.92
5	63.14	63.00
6	63.30	63.00
7	63.30	63.00
8	63.12	62.80
9	63.14	63.00
10	63.14	63.00
Average	63.20	62.94
Standard deviation	0.074	0.077

Table 4 contains the calculated standard deviations of differences between results of X-ray and gravimetric analysis for varying types of cement using pressed powder pellets. The corresponding standard deviation of wet gravimetric analysis for SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO, SO_3, and K_2O are included. Results are shown for samples prepared for X-ray analysis unground without internal standard correction, ground without internal standard correction, and ground with internal standard correction. It will be noted that the latter condition provides the accuracy of wet gravimetric analysis.

The precision or reproducibility of X-ray analysis of cements using pressed powder pellets with internal standard correction is shown in Table 5. Represented are the individual X-ray results for 10 successive determinations of two cements for CaO. The calculated standard deviation represents the measure of precision or reproducibility and is substantially less for the other element oxides.

Figures 1 through 7 show the calibration curves for cements ground with the use of internal standard correction. These curves are shown for all elements for which X-ray analysis has been readily used. It is important to note that all of the illustrated X-ray data use an external reference standard to correct for instrument fluctuations and varying conditions of the atmosphere during the time of analysis. Some instruments presently available perform this referencing or ratioing technique automatically. The particular instrument on which these data were developed requires manual manipulation of the reference standard and unknown to accomplish this ratioing technique.

The intensity ratio, shown as the ordinate of the calibration curves, represents the counting rates of the unknown sample to the standard reference sample, both of which have been corrected for internal standard variability. The internal standard serves as a monitor to provide for matrix correction, which may show either suppression or enhancement effects. The selection of internal standards must be such that the sample matrix affects the X-ray intensity of the element being measured and the internal standard element in equal and like extent. The intensity ratio may be represented by the following formula:

$$IR = \frac{UnA}{SRA} \times \frac{SRIS}{UnIS}$$

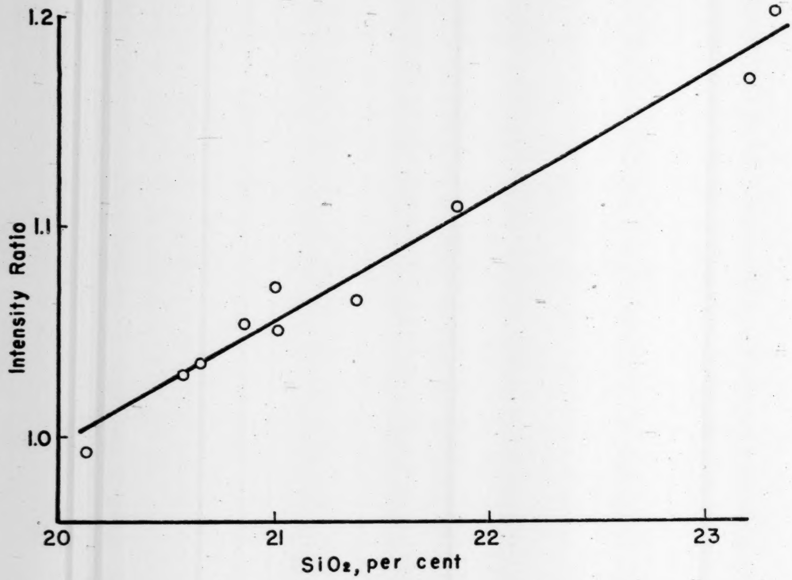

FIG. 1.—SiO_2 Calibration Curve for Cements Ground with Internal Standard Correction.

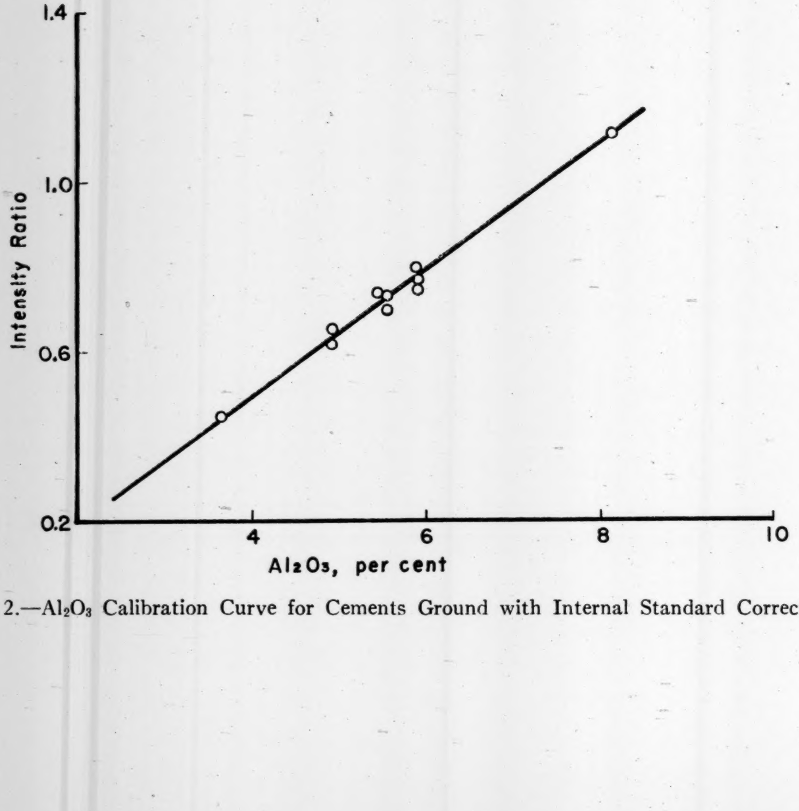

FIG. 2.—Al_2O_3 Calibration Curve for Cements Ground with Internal Standard Correction.

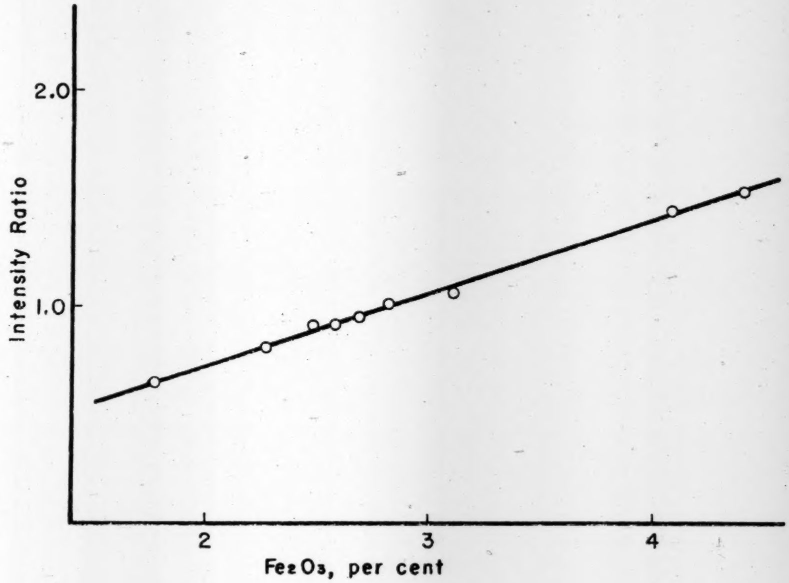

FIG. 3.—Fe_2O_3 Calibration Curve for Cements Ground with Internal Standard Correction.

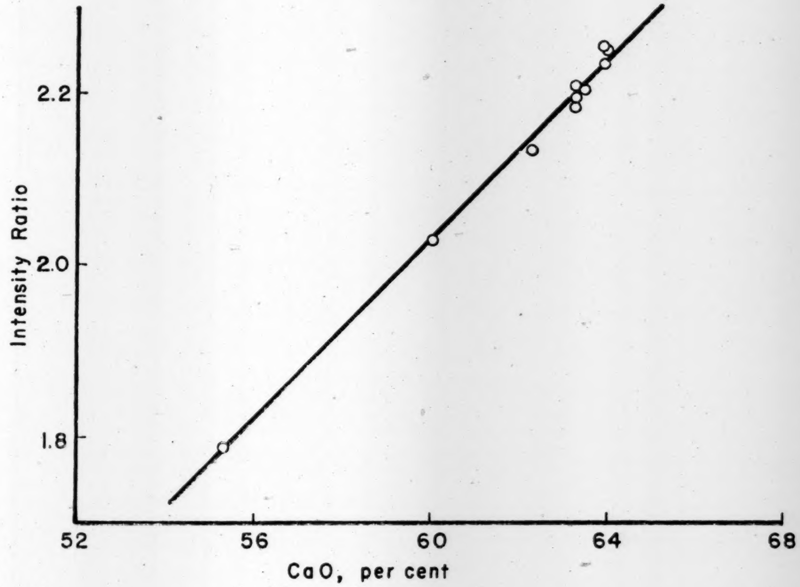

FIG. 4.—CaO Calibration Curve for Cements Ground with Internal Standard Correction.

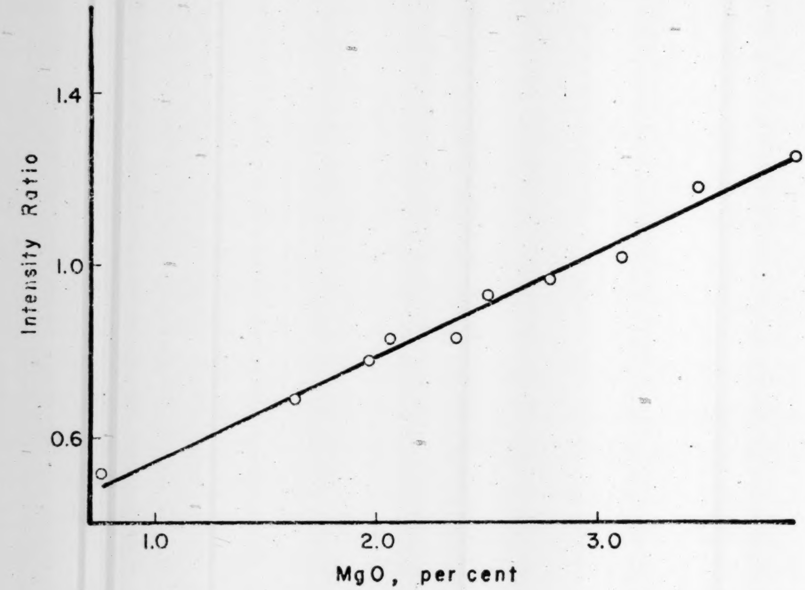

FIG. 5.—MgO Calibration Curve for Cements Ground with Internal Standard Correction.

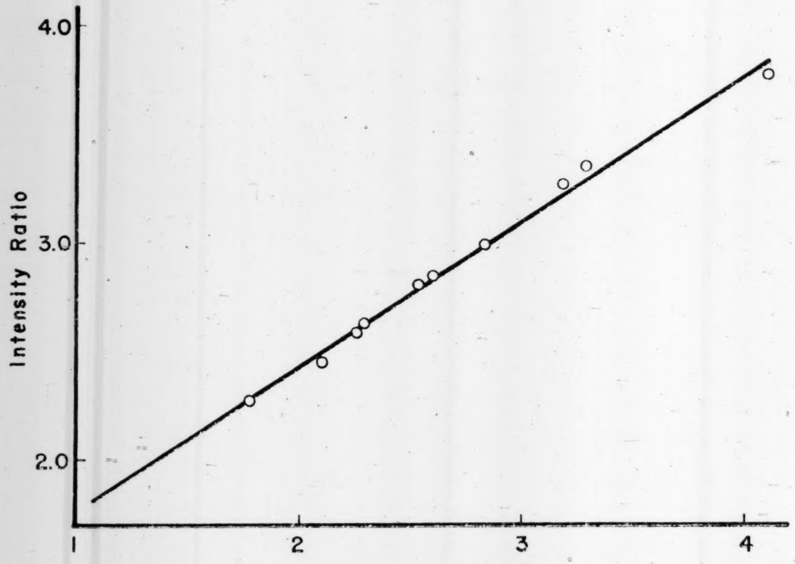

FIG. 6.—SO_3 Calibration Curve for Cements Ground with Internal Standard Correction.

where:

IR = the intensity ratio,

UnA = the counts per second of element *A* in the unknown sample,

SRA = the counts per second of element *A* in the standard reference sample,

SRIS = the counts per second of the appropriate internal standard element in the standard reference sample, and

UnIS = the counts per second of the appropriate internal standard element in the unknown sample.

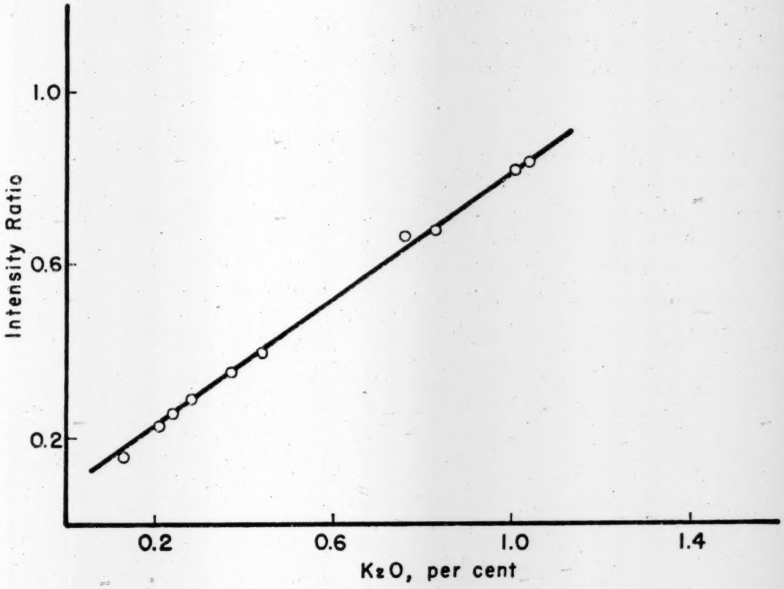

FIG. 7.—K_2O Calibration Curve for Cements Ground with Internal Standard Correction.

TABLE 6.—COMPARISON OF WET GRAVIMETRIC AND X-RAY ANALYSIS OF PLANT A RAW MIX USING PRESSED POWDER PELLETS.
Number of Raw Mix Samples, 8

	Standard Deviation of Differences Between Results of X-Ray and Gravimetric Analysis			
	SIO_2	AL_2O_3	Fe_2O_3	CaO
Samples unground	1.25	0.31	0.08	0.55
Samples ground	0.13	0.14	0.08	0.10

The determination of the accuracy and precision of X-ray emission spectroscopy techniques is greatly limited since the wet gravimetric methods must be relied upon to provide the accepted true value of analysis. While this has not been particularly troublesome in studies on cement because of the selection of samples on which many analyses were available and because the wet gravimetric methods for cement are well detailed and of proven reliability, it has been of major concern in the development of X-ray emission spectroscopy for raw material analyses.

The use of pressed powder pellets of raw materials that have been ground in the Bleuler mill with internal standards has provided a degree of accuracy and precision greatly surpassing the wet gravimetric methods. Such types of raw materials as sand, sandstone, clay, shale, marl, limestone and others yield a single calibration curve over the full range of elemental composition. It was on this phase of development that the major problems of particle size and matrix effects were first encountered and resolved.

The use of X-ray emission spectroscopy for raw mix control has been evaluated for a number of different plant locations. In cases where the raw mix is proportioned from a small number of relatively consistent raw materials, subsequent grinding of the raw mix has not been necessary, while for others, fine grinding is extremely important to achieve the accuracy required for plant process control. For example, the raw mix for one particular plant, plant A, is proportioned from as many as six and as few as two raw materials. There is good evidence that the form of the silica, whether it be flint from the limestone, quartz from sand, or silicates added in the form of shale, greatly affects the particle size distribution of the silica-bearing grains in the samples of raw mix. Fine grinding has reduced these to a consistent size and has therefore permitted precise chemical control.

Table 6 contains the calculated standard deviations of differences between results of X-ray and gravimetric analysis of plant A raw mix. The data represents the use of pressed powder pellets unground and ground, without internal standard correction, for SiO_2, Al_2O_3, Fe_2O_3, and CaO. MgO is not included as it does not constitute a control criterion at this location. The accuracy of X-ray results when the samples of raw mix are ground is equal to or better than that obtainable with wet gravimetric analysis.

SUMMARY

The use of pressed powder pellets is demonstrated to provide a simple, fast, and accurate method for the analysis of cement. These pellets eliminate the undesirable features in selection of internal standards and in time required for sample preparation and X-ray analysis that are associated with the use of fused pellets.

Particle size and matrix effects are eliminated by grinding the samples to $-20\,\mu$ with internal standards. Accuracy and precision are equal to those of wet gravimetric analysis when the ratioing technique of unknown to reference standard is applied under identical test conditions.

Raw materials and raw mix as well as cement can be analyzed by this technique.

For cement manufacturing plant process control, grinding is adequate for simple system analysis when only particle size effects are present. When neither particle size nor matrix effects are present, samples can be pelletized as obtained from the process. Individual process application must be used to determine the sample preparation procedure.

The technique of using pressed powder pellets, ground with internal standards, appears to have sufficient merit to warrant consideration by Committee C-1 for possible acceptance of X-ray emission spectroscopy as a tentative method for analysis of cement.

Acknowledgment:

The author is indebted to D. M. Schaefer, Chief of Chemical Section of the Marquette Research Laboratory, and to H. L. Austin, former Chief of Chemical Section, who performed the actual analyses and contributed to the development of X-ray application.

DISCUSSION

Messrs. C. V. McNicol[1] **and R. V. Neuman**[1] *(presented in written form)*.—Mr. Brown's paper has been of special interest to the South Pacific Division Laboratory as we have developed a similar method of routine testing of cement samples using a Phillips Electronics (Norelco) vacuum path X-ray emission spectrograph (**1–3**).[2] Tests were run using specially prepared external standards and pellets of cement mixed with starch or stearic acid. Matrix effects and difficulty of uniform mixing of the diluents caused considerable errors in the determinations. Simple briquetting of cement under high pressure using a similar cement as a standard seemed to give good results so a series of tests were run.

Instrument Settings:

The final instrument settings are shown in the accompanying Table 7. The 2θ settings for the elements and backgrounds were determined experimentally to correct for any misalignment of the instrument. The pulse height analyzer settings were all determined experimentally.

Sample Preparation Used in South Pacific Division Laboratory:

A 5.000-g sample of cement is placed in a $1\frac{1}{4}$-in. steel mold and No. 200 AB black bakelite mounting powder is placed on top as a backing material. The pellet is pressed at 30,000 psi with a slow release of pressure after the maximum is reached. The pellet is then placed face down on a piece of hard filter paper and the edges sprayed with Krylon plastic.

Test Results:

Routine testing of cement samples from a single mill was being done by standard wet chemical methods and eight of these samples were checked by the X-ray method using one of the previously analyzed cements as standard. Fixed counts were made on the samples and the standard for each setting of the instrument. Each element was corrected for background and then the percentage of each element was computed by using the ratio of counts per second for the unknown divided by the counts for the standard times the per cent of the element in the standard. Standard deviations were not computed, as only one wet chemical and one X-ray test were run on each sample, but a comparison of results is given in Table 8.

The average differences in the results are within the expected errors of routine wet chemical analyses. One X-ray result for calcium oxide seems to be too low by 0.64 per cent; this is unexplained.

Conclusions:

It is believed that the above described method will give adequate results for routine analysis of cements from one mill where variations are kept to a minimum. The grinding procedure described in Mr. Brown's paper should improve results, but our experience indicates that a binder additive is not necessary when a pressure of 30,000 psi is used.

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[1] Analytical Chemist, U. S. Army Engineer Division Laboratory, Corps of Engineers, South Pacific, Sausalito, Calif.

[2] The boldface numbers in parentheses refer to the list of references appended to this discussion.

TABLE 7.—INSTRUMENT SETTINGS ON PHILLIPS STANDARD LABORATORY VACUUM PATH SPECTROGRAPH FOR DETERMINATION OF CHEMICAL ELEMENTS IN PORTLAND CEMENT.

Tungsten target, vacuum path, and flow proportional counter with P-10 gas used for all elements The amplifier gain control was set at 10 and the voltage on the counter was 1645 v.

Element	Iron	Calcium	Silicon	Aluminum	Magnesium
Voltage, kv	20	20	35	35	35
Current, ma	15	15	35	35	35
Crystal	LiF	ADP	ADP	ADP	ADP
Peak position, deg 2θ	57.45	36.61	84.00	103.20	136.75
Number of counts	512 000	1 024 000	16 000	3200 to 6400	800
Background position, deg 2θ	59.00	37.50	85.00	105.00	138.80
Number of counts	2000	4000	4000	800 to 1600	400
Pulse height analyzer:					
Base line, v	...	...	23.4	18.0	10.8
Channel width, v	...	...	6.6	6.0	17.8

TABLE 8.—COMPARISON OF X-RAY SPECTROGRAPH AND WET CHEMICAL METHODS IN ROUTINE TESTING OF PORTLAND CEMENTS.

Sample	SiO_2, per cent			Al_2O_3, per cent			Fe_2O_3, per cent			CaO, per cent			MgO, per cent		
	X-Ray	Wet Chemistry	Difference	X-Ray	Wet Chemistry	Difference	X-Ray	Wet Chemistry	Difference	X-Ray	Wet Chemistry	Difference	X-Ray	Wet Chemistry	Difference
56–59	21.21	21.28	0.07	5.42	5.43	0.01	3.03	3.01	0.02	64.22	64.24	0.02	2.36	2.40	0.04
60–63	21.29	21.26	0.03	5.41	5.41	0.00	3.01	3.03	0.02	64.59	64.40	0.19	2.34	2.40	0.06
102–105	21.30	21.30	0.00	6.00	5.97	0.03	3.06	3.05	0.01	63.76	63.86	0.10	2.31	2.35	0.04
106–109	20.87	20.90	0.03	5.87	5.89	0.02	3.07	3.11	0.04	64.13	64.10	0.03	2.25	2.26	0.01
110–113	21.38	21.42	0.04	6.01	5.97	0.04	3.07	3.11	0.04	63.64	63.72	0.08	2.27	2.29	0.02
114–115	21.39	21.30	0.09	6.15	6.19	0.04	3.12	3.15	0.03	63.54	63.64	0.10	2.34	2.29	0.05
116–119	20.90	20.96	0.06	6.06	6.11	0.05	3.12	3.19	0.07	63.89	63.86	0.03	2.29	2.32	0.03
120–121	20.86	20.94	0.08	6.39	6.39	0.00	3.11	3.13	0.02	63.26	63.90	0.64	2.39	2.36	0.03
Average	...	...	0.05	...	...	0.024	...	...	0.03	...	...	0.15	...	...	0.04

MR. W. G. HIME.[3]—Claisse and Samson (4) recently suggested that particle size effects may be much greater when long-wavelength X-ray tubes are used. Does the author's finding that fine grinding eliminates particle size effects apply to both tungsten and chromium target X-ray tube sources?

MR. T. M. HOUSEKNECHT.[4]—How does the inclusion of an internal standard in grinding minimize or eliminate the matrix effect in exposed samples?

MR. L. U. SPELLMAN.[5]—Does the cellulose backing on the X-ray pellet cause undue loss of time in out-gassing? Will the Bleuler mill take only a few grams of charge as indicated by the use of cellulose backing?

MR. B. L. BEAN.[6]—What was the source of the sodium lauryl sulfate used as a grinding aid? Was it of reagent grade or was it a commercial product such as is used in laundering? Was it necessary to correct the SO_3 results obtained for the amount of sulfur present in this grinding aid?

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[3] Supervisor, Analytical Research Laboratories, Portland Cement Assn , Skokie, Ill.

[4] Atlantic Chemical Co., Inc., Ravena, N. Y.

[5] Manager, Technical Services and Distribution, Atlantic Cement Co., Inc., Ravena, N. Y.

[6] Chemist, Inorganic Building Materials Section, National Bureau of Standards, Washington, D. C.

How was it determined that no particles larger than 20 μ were present after grinding for 6 min—by microscopic examination or some other means?

MR. O. E. BROWN (*author's closure by letter*).—Messrs. McNicol and Neuman have apparently made a considerable study of the analysis of cement by X-ray emission spectroscopy. I concur with the conclusions that their method gives adequate results for routine analysis of cements from one mill where variations are kept to a minimum. However, use of fine grinding and internal standards has proved more convenient for laboratories analyzing a variety of cement types or cements from more than one mill. A single calibration curve is certainly to be preferred over a multitude of separate curves for each type of cement for each mill.

A point that should be clarified is that a binder additive is not employed in the method proposed. The sodium lauryl sulfate is a grinding aid to prevent compaction of material in the mill. Cellulose powder is used as a backing material to increase pellet thickness for added rigidity. The pressed cellulose backing also provides a convenient surface for pellet identification.

Mr. Hime's question was unanswerable at the time the paper was presented. It can now be said that the fineness of grind achieved with the proposed method is equally suited to the use of tungsten and chromium target X-ray tubes.

Mr. Houseknecht's question pointed out a weak point in the originally distributed paper. The properly selected internal standard will be affected by sample matrix in a manner similar to the element of interest. The internal standard can thus serve as a monitor for matrix correction.

Mr. Spellman asked if the cellulose backing causes undue loss of time in out-gassing. Experience has indicated that from 1 to 3 min are required to attain the operating vacuum level with a four-position chamber.

Mr. Spellman also inquired of the capacity of the Bleuler mill. The mill will grind up to 50 g of cement sample. However, grinding time to achieve a given level of fineness increases as the mill charge increases. The proposed sample size is the minimum practical to attain the required fineness in shortest time, all factors considered.

In reply to Mr. Bean's question on the source of the sodium lauryl sulfate, the purest grade available, Matheson, Coleman, and Bell DX2490 (95 per cent), was used. The calibration curve for SO_3 is drawn without correction for the amount of sulfur present from the grinding aid. Such correction would merely change the relative position of the X axis to the Y axis since it would be constant.

Maximum particle size was measured by both microscopic and Coulter counter techniques on ground samples. No particles larger than 20 μ were detected, although actual measurement of top size was not attempted.

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(3) George Andermann, *Analytical Chemistry* Vol. 33, No. 12, 1961, p. 1689.

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CONTROL OF FALSE SET BY THE USE OF ANHYDRITE AND GYPSUM BLENDS*

BY JAMES L. SAWYER[1]

False set in portland cements, otherwise known as premature stiffening or grab set, may be a troublesome characteristic in many modern cements. This is especially true in fine-ground type III products. With this type of set the rapid stiffening of neat pastes, mortars, and in extreme cases concrete mixes, occurs as soon as the mixing procedure has been completed and the batch allowed to stand undisturbed a few minutes. Reworking the stiffened material to its original workability is usually possible without the addition of extra mixing water. In extreme cases a small amount of additional mixing water is required to restore the batch to a plastic condition. Once remixed, after full development of the false set, no further stiffening occurs until the beginning of the true setting and hardening action. This type of set differs from quick or flash set in that no appreciable amount of heat is liberated, and reworking has only a minor effect on early strength development.

Various theories have been enumerated by Hansen (**1**)[2] as having been considered over the years as possible causes of false set. However, the generally accepted theory today is that false set is caused by the presence in the cement of either calcium sulfate hemihydrate or soluble anhydrite, or both, which upon the addition of water dissolve rapidly and precipitate as gypsum (**2**). It is believed that due to the heat of grinding and the conditions during storage of the cement, the gypsum dehydrates to form the hemihydrate and perhaps soluble anhydrite. These, with the addition of mixing water, set up very rapidly to produce false set. As only a small amount of the total mixing water is required for this recrystallization of gypsum, the stiffened mix can be reworked to a plastic condition with little loss of workability.

This theory is thought by some to explain not only the false set occurring in fresh cement but also that occurring only after aeration of a cement not normally false setting. As stated by Steinour (**3**): "When false set resulting from hot milling does not occur at once but only after aeration of the cement, this may be owing to initial high reactivity of the tricalcium aluminate (C_3A) sufficient to combine sulfate at a rate that prevents marked supersaturation with gypsum. The aeration is thought to decrease the reactivity of the tricalcium aluminate."

Still another type of rapid stiffening due to aeration has been reported by

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

Swayze (4,5). Here tests were made using a clinker that contained no C_3A and little or no gypsum. The rapid stiffening induced by aeration of this cement could, therefore, not be attributed to inactivation of C_3A or dehydrated gypsum, as both materials were absent. This type of rapid stiffening should perhaps be referred to as quick set since it could not have been caused by the crystallization of gypsum.

Swayze offers two possible explanations for this type of rapid stiffening due to aeration. He proposes that the activation of cement grains by absorption of or combination with very thin films of water causes these surfaces to combine rapidly with water, and a gelling action therefore develops rapidly. Another explanation for the type of quick set induced by aeration and also false set in fresh cements in which gypsum has been converted to hemihydrate by the heat of grinding, was proposed by Swayze in 1960 (5).

In this theory he considered the possibility that moisture during aeration, and perhaps the moisture liberated by the dehydration of gypsum, prehydrates the C_3S grains, thus liberating very fine-grained and quickly soluble calcium hydroxide. This liberated calcium hydroxide causes flocculation of the cement grains and thus the rapid stiffening of the cement paste.

USE OF ANHYDRITE

As has been stated by Hansen (6): "The use of calcium sulfate to prevent flash set led to the development of what has become known as false set." However, Hansen also stated (7): "Calcium sulfate is unique in that it is an abundant natural product that has the ability to modify the reaction that produces flash set in portland cement, accelerate the rate at which cement develops its strength, and favorably affect the volume change characteristics of the product produced from the cement."

In an early investigation Berger (8) found that the use of mixed retarders (anhydrite and plaster or gypsum and plaster) produced a more satisfactory cement than was obtained by the use of any one form of calcium sulfate. However, he did not feel that anhydrite could be used alone. Meade (9) stated, in 1928, that anhydrite is an effective retarder for cement clinker. He concluded that the active retarding constituent is SO_3 and, therefore, anhydrite is desirable because of its high SO_3 content.

Later researchers have pointed out that natural anhydrite dissolves much more slowly than gypsum and thus, in spite of a higher solubility than gypsum, can often give the effect of lower solubility. It would appear, therefore, that gypsum with its faster solubility would give greater protection from flash set than would anhydrite. W. C. Hansen, in a personal communication points out, "We should not speak of the solubility of either anhydrite or hemihydrate in water at normal atmospheric temperatures. These compounds are unstable in such a system and form solutions that are generally metastable and supersaturated with respect to gypsum, which is the stable phase." Kondo (10) has recommended that the gypsum addition be limited to that necessary to give 1 per cent SO_3 in the cement, and that anhydrite be added to bring the SO_3 content up to the optimum amount. Kuznetsov (11) has observed that the addition of anhydrite gave cements of higher strengths and durability. Hansen and Hunt (12) found that a mixture of gypsum and anhydrite gave good retardation and did not adversely affect the other physical properties of the cement. Webb and Moody (13) concluded that natural anhydrite was a safe and effective retarder for clinker produced at one mill having a low C_3A and alkali content. Steinour (14) concluded that natural anhydrite is a suitable retarder for some clinkers, and that mixtures of natural anhydrite and gypsum are suitable for use with many clinkers. He pointed out that if the gypsum is sufficient to take care of the initial rapid reaction of C_3A, the anhydrite suffices for the further, slow reaction.

It would appear then that cements low in C_3A content do not need gypsum additions to retard set, and thus can stand complete replacement of gypsum by anhydrite. Higher C_3A clinkers would require increasing amounts of gypsum to control set and therefore cannot stand more than partial replacement of gypsum by anhydrite.

FALSE SET IN A SPECIAL TYPE III CEMENT

The problems of false set are not unique with any one producer or type of cement. It is, however, of particular concern where the high fineness required for type III cement is conducive to high grinding temperatures. This problem became a particular nuisance when the Lone Star Research Laboratory at Hudson, N. Y., evaluated a special type III cement for its application in concrete construction. This cement had a zero tricalcium aluminate content and high early-strength properties and had shown outstanding performance, in comparison with types II and V cements, in its ability to withstand strong sulfate waters.

Therefore, it was felt that this cement would be applicable for concrete construction in areas with strong sulfate soils and waters. Upon checking its concreting properties, it was found that this cement unfortunately had a decided tendency to give a false set.

To overcome this difficulty, the Hudson Research Laboratory began a study of retarder influence on false set of this cement. Tests on laboratory grinds led us to recommend the use of anhydrite in place of gypsum for this cement. This cement had no tricalcium aluminate and was low in alkalies, and although Kondo (15) has pointed out the iron phases set almost as rapidly as C_3A, gypsum was not required to prevent flash set. Thus an ideal situation existed for the control of false set by the use of natural anhydrite.

EFFECT OF ANHYDRITE ON CONCRETING PROPERTIES

Concrete tests on representative samples of this type III cement produced using anhydrite as a complete replacement for gypsum showed a complete absence of either false set or quick set. Even overnight aeration of this cement in a moist atmosphere failed to induce any rapid stiffening in a rich-mix concrete. The false set according to ASTM Method of Test C 359[3] was better than with gypsum; however, some stiffening, which might have indicated a very mild quick set, did occur during this test. In these mortar tests, 32 per cent water was used, corresponding to a water ratio of 3.6 gal per sack of cement. This corresponds to a water ratio of 4.6 gal per sack for a 7.5 sack concrete mix with 2-in. slump. Since rate of development of stiffening must be a function of the original paste density, the only explanation for complete absence of quick set in concrete with water ratios of 4.6 gal per sack or higher for a period of 12 to 15 min and development of a quick set in the standard mortar within 11 min or less is that the test method is too severe for practical use. We believe that any cement which shows a moderate or high penetration for the mortar after 5 min is above suspicion so far as its action in concrete is concerned.

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[3] Method of Test for False Set of Portland Cement (Mortar Method) (C 359), 1961 Book of ASTM Standards, Part 4, p. 139.

Table I gives data on concrete tests made on this cement. In addition to the cement produced with 100 per cent gypsum and 100 per cent anhydrite, other production runs were made using 25 per cent gypsum: 75 per cent anhydrite and 50 per cent gypsum: 50 per cent anhydrite by weight. The reasons for using a blend of gypsum and anhydrite rather than either alone will be discussed in detail later.

TABLE I.—CONCRETE TESTS.

Sacks per cu yd	Water: Cement Gal per sack	Slump, in.			Compressive Strength, psi			
		1st	2nd	Loss	1 Day	3 Days	7 Days	28 Days
			100 PER CENT GYPSUM					
3.63.......	9.89	2.3	[a]	...	970	1670	2310	2940
4.55.......	7.52	2.2	[a]	...	1630	2730	3650	4420
6.04.......	5.48	2.2	[a]	...	2900	4700	5570	6450
7.53.......	4.48	1.9	[a]	...	3540	5510	6490	7900
			100 PER CENT NATURAL ANHYDRITE					
3.58.......	9.67	1.9	1.9	0.0	1030	1960	2460	3020
4.51.......	7.23	2.0	1.9	0.1	1780	3220	3850	4600
6.00.......	5.38	2.0	2.1	+0.1	2900	4460	5260	6040
7.49.......	4.59	2.1	1.7	0.4	3600	5480	6140	7310
			25 PER CENT GYPSUM: 75 PER CENT ANHYDRITE BY WEIGHT					
3.62.......	9.67	2.1	2.0	0.1	970	1800	2390	3140
4.53.......	7.25	2.0	1.9	0.1	1640	2860	3680	4990
6.03.......	5.42	2.0	1.8	0.2	2820	4640	5220	6430
7.51.......	4.51	2.0	1.5	0.5	4020	5480	6280	7100
			50 PER CENT GYPSUM: 50 PER CENT ANHYDRITE BY WEIGHT					
3.60.......	9.18	2.0	1.3	0.7	1440	2510	2980	3380
4.54.......	7.13	2.0	1.2	0.8	2120	3620	3940	4820
6.07.......	5.30	2.0	1.3	0.7	3550	5390	6000	6810
7.56.......	4.51	2.0	1.3	0.7	4430	6470	6660	7460

[a] Tests omitted due to rapid development of false set.

Table I shows concrete tests made using four different concrete mixes designed to contain 3.6, 4.5, 6.0, and 7.5 sacks of cement per cu yd. All mixes were made using siliceous sand-gravel mixes graded up to 1½-in. gravel size. Compressive strength tests were made on moist-cured 6 by 12-in. cylinders. Two slump tests were made on each batch—the first immediately after completion of mixing and the second after the concrete had stood for 5 min in an open pan. Concretes displaying false set generally show losses of 1 in. or more, for 2-in. slump concrete, between the time of making the first and second slump determinations.

As has been previously stated, gypsum alone gave a cement with a decided false set. This was pronounced enough for this particular sample so that the second slump had to be omitted due to the rapid stiffening. However, the cement produced using 100 per cent anhydrite shows practically no change in slump during this period. This retention of plasticity is about the same for the blend of 25 per cent gypsum: 75 per cent anhydrite. However, when more gypsum was used as in the case of the 50 per cent gypsum: 50 per cent anhydrite blend, a slight tendency to stiffen could be detected.

TABLE II.—STANDARD PHYSICAL TESTS.

	100 per cent Gypsum	100 per cent Anhydrite	25 per cent Gypsum: 75 per cent Anhydrite	50 per cent Gypsum: 50 per cent Anhydrite
Fineness, Passing No. 325 Sieve	99.3	98.8	99.5	99.3
Fineness, Wagner	2750	2790	2980	2870
Fineness, Blaine	4480	4460	4720	4480
Normal Consistency	26.5	25.5	26.0	26.5
Gilmore Set, Initial	2:00	2:40	2:35	2:30
Gilmore Set, Final	4:20	4:50	4:35	4:15
Air, per cent	4.8	7.1	7.1	9.1
Tensile Strength, psi:				
1 Day	265	270	305	335
28 Days	575	525	560	530
Water for Mortars, per cent	45.0	46.5	46.0	47.0
Compressive Strength, 2-in. Cubes, psi:				
1 Day	2440	2230	2850	2710
28 Days	7550	7080	7660	7830
Flexural Strength (C 348), psi:[a]				
1 Day	470	555	565	620
28 Days	1310	1185	1145	1095
Compressive Strength (C 349) psi:[b]				
1 Day	2630	2390	2780	2980
28 Days	7920	7290	7630	7260

[a] Method of Test for Flexural Strength of Hydraulic Cement Mortars, 1961 Book of ASTM Standards, Part 4, p. 165.

[b] Method of Test for Compressive Strength of Hydraulic Cement Mortars (Using Portions of Prisms Broken in Flexure), 1961 Book of ASTM Standards, Part 4, p. 135.

It would appear from these data that either the 100 per cent substitution of anhydrite for gypsum or the 25 per cent gypsum: 75 per cent anhydrite blend will produce an excellent type III cement that is entirely free of false set in concrete mixes.

PHYSICAL PROPERTIES

In Table II we can observe results of standard physical tests on these cements.

These data show that the cement produced, using natural anhydrite as a total replacement for gypsum, gave results comparable to that obtained when gypsum alone was used. However, in both cases the early tensile strengths are lower than desired. The addition of a blend of 25 per cent gypsum: 75 per cent anhydrite gave a higher 24-hr tensile strength than did either retarder alone, and, as has been previously noted, this small amount of gypsum did not cause premature stiffening. It was, therefore, felt that the use of a retarder blend composed of 25 per cent gypsum and 75 per cent natural anhydrite, by weight, was ideal. This blend gave a cement that was free of false set, had high early strength, and was somewhat easier to grind than cement produced with anhydrite alone.

FINENESS CONSIDERATIONS

The higher specific gravity and greater hardness of anhydrite over gypsum causes a difference in ratio of Wagner and Blaine finenesses when cements are ground with one or the other of these two retarders.

The following data (16) show average Wagner and Blaine finenesses obtained on laboratory grinds with the two retarders:

Retarder	Specific Gravity	Hardness, Mohr's Scale	Fineness		
			Wagner	Blaine	Ratio
Gypsum........	2.32	1.5 to 2.0	2660	4500	1.69
Anhydrite.......	2.90	3.0 to 3.5	2720	4250	1.56

As is well known, the Wagner surface area is based on two false premises: it assumes that all particles in the cement have the same specific gravity and the same ability to transmit light. In the case of a soft, low specific gravity material, such as gypsum, all of it is ground very finely, and most of it is concentrated in the $-7.5\ \mu$ range at the time of the final reading, although many particles may be up to $10\ \mu$ in size. While the higher light transmittancy of gypsum over clinker particles tends to compensate for their slower settling rate, Wagner surfaces are usually higher than they should be when percentages of gypsum in a cement are high.

With the specific gravity of the anhydrite being so close to that of clinker, the settling rate of anhydrite particles will be faster than that of gypsum particles, and a closer indication of fineness, as determined by the Wagner method, will be obtained for cements containing anhydrite in place of gypsum.

Blaine finenesses for cements with high gypsum content will be higher than they should be, unless the formula which corrects for bed porosity and specific gravity is used. Blaine finenesses for anhydrite cements will be on the low side, unless corrections for bed porosity and specific gravity are made.

Our experience indicated that for cements in which anhydrite replaced all or part of the gypsum, a higher Wagner fineness was required to maintain the same 24-hr strength. A slight reduction in Blaine fineness as determined by the simple time formula could be permitted without loss of early strength.

MILL PROBLEMS

Some mill problems are introduced by the substitution of anhydrite for gypsum in finish grinding. Gypsum acts as a good grinding aid, while anhydrite does not.

When anhydrite was used to replace a major portion of the gypsum, it was observed that the desired fineness could not be maintained with the usual separator settings. Production rates suffered when the desired fineness was maintained. Also, some mill grate plugging occurred. These delays and a lower grinding rate can—and did at one mill—cause a serious imbalance between kiln and finish-mill capacity. Therefore, it became imperative that steps be taken to improve the grinding rate when anhydrite was used.

Table III shows the experience at one mill over a period of several years. As we can see from these data, the use of anhydrite even as a partial replacement for gypsum reduced the grinding rate by nearly 20 per cent. In fact during some production runs the reduction was as high as 24 per cent.

Fortunately, the addition of water to the finish grinding mills improved grindability and also helped raise the Blaine fineness. When the water addition was increased to 0.767 per cent the grinding rate and the ratio of Blaine and Wagner fineness approached those obtained with gypsum alone.

Blending of the two retarders was originally achieved by using a payloader to dump one bucket of gypsum to two buckets of anhydrite on the ground. This was blended and then fed to the mills. This procedure was costly in man-hours and perhaps led to inaccuracies in proportioning. This system has since been replaced by separate storage facilities for gypsum and anhydrite, both of which are equipped with feeders. Hence the percentage of each can be controlled very closely.

TABLE III.—EFFECT OF RETARDER AND MOISTURE ON GRINDING RATE.

	1957	1958	1959	1960	June 1961
Barrels per hr	59	48	49	52	58.4
Gypsum, per cent	100	25	25	25	25
Anhydrite, per cent	0	75	75	75	75
Water, per cent	0	0	0	0.3	0.77
Wagner	2810	2850	2870	2890	2840
Blaine	4790	4670	4700	4760	4820
Ratio	1.70	1.64	1.64	1.65	1.70

USE OF ANHYDRITE IN CEMENTS OTHER THAN SPECIAL TYPE III

As previously stated, our interest in the use of anhydrite was originally confined to a special type III cement which was produced at two Lone Star mills. One of these mills has been using anhydrite successfully since 1958; the other mill first used this retarder in 1959 and its operation has been equally satisfactory.

One mill has used natural anhydrite in the production of special type III cement only. The second mill has used anhydrite successfully for up to 83 per cent of the SO_3 in this special type III cement, up to 30 per cent in regular type III cement, and up to 50 per cent in type I and type II cement.

CONCLUSIONS

1. A literature review showed that false set is caused by the dehydration of gypsum during grinding and subsequent storage, according to the universally accepted theory.

2. The literature review, experimental grinding, and extended production show that a logical solution to the problem of false set is the use of anhydrite as a partial replacement for gypsum.

3. False set was eliminated by the use of anhydrite at two of the Lone Star mills.

4. Complete substitution of anhydrite for gypsum was not satisfactory due to low early strength and reduced grinding capacity.

5. A blend of 25 per cent gypsum and 75 per cent anhydrite, by weight, appears to be the most satisfactory combination to eliminate false set and give satisfactory strength and grinding capacities.

6. The most notable improvement in grinding capacity was achieved when water was added in approximately the same amount that had been lost due to the substitution of anhydrite for part of the gypsum.

REFERENCES

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(2) W. C. Hansen, "False Set in Portland Cement," *Proceedings*, Fourth International Symposium on the Chemistry of Cement, Vol. I, p. 388 (1960).

(3) H. H. Steinour, "The Setting of Portland Cement—A Review of Theory, Performance and Control," *Bulletin 98*, Research Laboratories of the Portland Cement Assn., p. 92 (1958).

(4) M. A. Swayze, Discussion of W. C. Hansen, "Aeration Cause of False Set in Portland Cement," *Proceedings*, Am. Soc. Testing Mats., Vol. 58, p. 1050 (1958).

(5) M. A. Swayze, Discussion of W. C. Hansen, "False Set in Portland Cement," *Proceedings*, Fourth International Symposium on the Chemistry of Cement, Vol. I, p. 417 (1960).

(6) W. C. Hansen, "Aeration Cause of False Set in Portland Cement," *Proceedings*, Am. Soc. Testing Mats., Vol. 58, p. 1044 (1958).

(7) W. C. Hansen, "The Role of Calcium Sulfate in the Manufacture of Portland Cement," *Publication No. 63*, National Ready Mixed Concrete Assn., March, 1956.

(8) E. E. Berger, "Calcium Sulphate Retarders for Portland Cement," U. S. Bureau of Mines, *Technical Paper 451* (1929).

(9) R. K. Meade, "Manufacturing Conditions Influencing Setting Time of Portland Cement," *Rock Products*, March 31, 1928.

(10) R. Kondo, "False Setting of Cement," *Yogyo Kyokai Shi* (Journal of the Ceramic Assn., Japan), Vol. 65, p. 211 (1957).

(11) A. M. Kuznetsov, "Increasing the Quality of Portland and Clinker - Portland Cements by the Addition of Natural Anhydrite," Prom. - Ekon., *Byulletin*, Sovet. Narod., Khoz. Perm. Ekon. Admin. Raiona, No. 9, p. 17 (1958).

(12) W. C. Hansen and J. O. Hunt, "The Use of Natural Anhydrite in Portland Cement," ASTM BULLETIN, No. 161, p. 50–58, Oct., 1949.

(13) S. B. Webb, Jr. and J. W. Moody, "Regulation of Set in Portland Cement by Anhydrite," *Mill Session Paper M-152*, Research and Development Division of the Portland Cement Assn., p. 8 (1958).

(14) H. H. Steinour, "The Setting of Portland Cement—A Review of Theory, Performance and Control," *Bulletin 98*, Research Laboratories of the Portland Cement Assn., p. 90 (1958).

(15) R. Kondo, Discussion of W. C. Hansen, "False Set in Portland Cement," *Proceedings*, Fourth International Symposium on the Chemistry of Cement, Vol. I, p. 411 (1960).

(16) M. A. Swayze, "Report on Study of Causes of False Set in Cements," Unpublished Report, Lone Star Cement Corp., May, 1958.

(See Discussion on page 926.)

DISCUSSION

MR. M. A. SWAYZE[1] (*presented in written form*).—Mr. Sawyer's paper is largely based on experiences with use of anhydrite as a substitute for gypsum in a zero C_3A type III cement produced at the West Texas Lone Star plant, first in laboratory research at Hudson, N. Y., early in 1958, and later in mill production at the plant. The research grinds at Hudson included grinding of the basic clinker with no additions and with enough gypsum, commercial plaster of Paris, or anhydrite to yield 2.5 per cent SO_3 in the cements. All grinds were made in duplicate at room temperature to type III fineness, then stored in sealed cans. Laboratory tests of these four cements included false set tests by the mortar method, loss on ignition, water requirements and flows in Method C 109[2] mortar and compressive strengths at 1, 3, 7 and 28 days. Progress in grinding was included in the report.

Subsequent treatment of each sample included the following: (1) aeration of freshly ground cements in laboratory overnight, (2) heating of the fresh samples at 260 F, 7 days in open cans, and (3) aeration of heated samples overnight in laboratory air. False set tests, together with loss on ignition and strength tests were repeated for each condition of exposure. Subsequent grinds were made with small water additions to the straight clinker, clinker plus gypsum, and clinker plus anhydrite mixtures. Other grinds were made with the three sulfates, with Ottawa sand substituted for the clinker. There were no instances whatever in the tests for false set where the Method C 359[3] mortar developed any sensible heat or where remixing failed to yield full penetration of the needle in the test. Hence we could not characterize the behavior of any sample as anything except false set under the old definition of this property.

This research required three months of active work, and was under the active supervision of Mr. Sawyer. The writer made frequent visits to Hudson during this period in order to be thoroughly familiar with all phases of the work. Our report on these studies was made on May 16, 1958, for use by the Lone Star operating staff. A complete copy of the report was given within a week after completion to W. C. Hansen, Chairman of the Working Committee on False Set of ASTM Committee C-1 on Cement. The report in no part gave any indication of quick setting but only the development of false sets. A very brief summary of the report, omitting the data on cement grinds containing the hemihydrate addition, was given as a discussion of Hansen's paper[4] at the 1958 ASTM Annual Meeting regarding the cause of false set in portland cements by aeration.

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[1] Cement Consultant, Scarsdale, N. Y.

[2] Method of Test for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. Cube Specimens) (C 109), 1961 Book of ASTM Standards, Part 4.

[3] Method of Test for Compressive Strength of Thermal Insulating Cement (C 359), 1961 Book of ASTM Standards, Part 5.

[4] W. C. Hansen, "Aeration Cause of False Set in Portland Cement," *Proceedings*, Am. Soc. Testing Mats., Vol. 58, 1958, p. 1044.

In spite of lack of any evidence in our 1958 report to support the statement that quick setting was found for the grind made without any retarder, Mr. Hansen stated in his paper at the 1960 International Symposium on Cement in Washington that, "Swayze showed that aeration caused flash set with a clinker that contained no calculated C_3A and assumed that aeration increased the reactivity of C_3S." I was compelled to refute this false statement in a discussion of his paper, and offered a suggestion that study of the colloidal behavior of fresh cement pastes might well prove valuable in explaining false set from any cause, as was originally suggested by L. S. Brown while engaged in research at the Lone Star Laboratory during 1940.

In spite of this, Mr. Hansen persisted in his contention that quick setting had occurred upon aeration of this cement in his paper, "Quick and False Set in Portland Cements" as published in the October 1961 issue of *Materials Research & Standards*. In this issue he stated, "Swayze found that this (zero C_3A) clinker, when ground with gypsum, became quick setting on aeration just as it did when ground without gypsum." This statement was even more false than the one in his symposium paper, but could not be refuted by me, since after spending 7½ months in Great Britain, on my return home in October, 1961 I was confronted by such a stack of urgent mail that I could not get to read his article until long after any critical comment would have been effective.

The present paper by Mr. Sawyer, however, is quite another matter. I was so shocked at the several references to "quick" set of samples in our 1958 research series, that I went to Hudson especially to see him. On reviewing the reasons for these references to "quick" set, I found that in each case he still believed what we had dealt with was false set rather that quick set, if we considered false set in the broader aspects of the term. However, as a member of the Subcommittee on Papers and Symposia of Committee C-1 on Cement, Mr. Hansen had reviewed his paper and suggested (but not demanded) that, with the new definition of false set now under consideration being limited to the hardening of hemihydrate into gypsum in a cement paste as the sole cause of false set, several of Sawyer's references to false set would be beyond the pale of the new false set definition, and might therefore well be changed to "mild quick set" or some similar term. In deference to Hansen's suggestions, these changes were made.

My quarrel with these changes is not so much with Mr. Hansen as it is with the very narrow and biased definition of false set that the Working Committee on False Set has brought forth. The Lone Star Cement research has clearly indicated that there is more than one cause of false set, if the transition from hemihydrate to gypsum in a fresh neat paste is considered as one of them. As the Lone Star research in 1958 brought out, the action in each case, regardless of composition or exposure, was exactly the same—an originally plastic mortar stiffened more or less rapidly with no perceptible evolution of heat and then returned to a plastic condition again on remixing, which a real quick-setting cement cannot possibly do. Since in the Lone Star tests on clinker without gypsum the setting of the cement mortars was so greatly prolonged that 24-hr compressive strengths were less than 10 per cent of normal strengths of the cements containing sulfate, the behavior of the pure clinker grinds was the very antithesis of real quick set.

In my estimation Committee C-1

would be extremely foolish to adopt any such narrow definition of false set as Mr. Hansen transmitted to Mr. Sawyer. It would most effectively kill off any further research to discover the true basic cause or causes for false set, if there happens to be more than one. It is possible even that the transition from hemihydrate to gypsum in a fresh cement paste is not so much due to a "plaster set" as it is to a very strong flocculation of the paste particles by the hemihydrate. There is some good evidence of this possibility in the records of grinding rates in the Lone Star 1958 data. Consider the following information on laboratory mill revolutions needed to attain 2700 Wagner fineness in that report:

Cement Composition	Cement Grinds, Mill Revolutions		Sand Grinds, Revolutions, No Water Added
	No Water	Water Added	
Clinker alone.........	20 000	8 000	...
Clinker + gypsum....	7 000	8 000	9 000
Clinker + plaster.....	50 000	...	16 000
Clinker + anhydrite..	18 000	8 000	8 000

In the 1940 work done by L. S. Brown, gypsum was found to be strongly negative electrostatically, which is why it is such a good grinding aid. The same holds for rosin and other grinding aids. Since the hemihydrate is such a great deterrent to grinding, it could easily be that it is just as strongly electropositive, which would account for its great interference with grinding, the very rapid development of coating in the grinding mill, and therefore the greatly lowered mill efficiency, as well as the marked flocculation to be found in false set pastes. In colloid phenomena, electrically charged particles generally act the same way in liquid suspensions as in air, provided the liquid does not change their composition and therefore the nature of their charge. We know that silica sand has a negative electrostatic charge during grinding, which is why it separates so nicely from the positively charged calcium carbonate if the mixture is free from interfering fines, when run through the Johnson electrostatic separator. Brown found C_3S grains to be either neutral or slightly electronegative. No work was done on cement grains, which are a composite of the cement compounds. At any rate, this matter of colloid chemistry and the chances for its great influence on the character of fresh cement pastes must be worked out before any arbitrary and restrictive definition is placed on the cause of false set in portland cement.

MR. JAMES L. SAWYER (*author's reply by letter*).—I would like to thank Mr. Swayze for his discussion and to thank him especially for calling attention to the question of the proper use of the terms "false set" and "quick set."

In every instance the rapid stiffening to which I refer in my paper occurred without sensible heat liberation, and plasticity was restored by remixing.

If the definitions as given in ASTM Methods C 359 and C 451[5] are the terms thought to be most correct, then the only place in my paper where the term "quick set" should appear is in the first paragraph.

If, however, the definitions recommended by the C-1 Working Committee on False Set (as given in Mr. Gilliland's paper on "Significance of False Set Tests"[6]) are accepted, then obviously any rapid stiffening not possibly caused by the crystallization of gypsum must be referred to by some term other than "false set."

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[5] Method of Test for False Set of Portland Cement (Paste Method) (C 451), 1962 Supplement to Book of ASTM Standards, Part 4.

[6] See p. 880.

In writing this paper I attempted to conform to these new definitions.

MR. J. L. GILLILAND.[7]—Mr. Sawyer's reference to "quick set" rather than "false set" in the special cement he describes is based on recent actions by the Working Committee on False Set to revise the "definitions" and "interpretations" in Methods C 359 and C 451. These revisions will be presented to Committee C-1 during the December 1963 meeting. They are given in detail in my paper on the "Significance of the False Set Tests.[6]"

The general discussions on false set, following the presentations at this meeting, point out the different viewpoints represented. If false set is defined simply as a condition which can be removed by reworking, then cases of mild quick set are classed as "false set." On the other hand, when false set is defined as resulting from the crystallization of gypsum, those instances where no sulfate is present cannot be called false set. No doubt there always will be exceptions to any definitions, but the Working Committee feels that these new definitions are more satisfactory.

This discussion brings to mind the conversation between Humpty Dumpty and Alice in Lewis Carroll's *Through the Looking Glass*:

> "When I use a word," Humpty Dumpty said, in rather a scornful tone, "it means just what I choose it to mean—neither more nor less."
>
> "The question is," said Alice, "whether you *can* make words mean so many different things."
>
> "The question is," said Humpty Dumpty, "which is to be master—that's all."

MR. BRYANT MATHER.[8]—Mr. Sawyer reports that gypsum is a grinding aid and anhydrite is not. He further finds that anhydrite plus water works essentially as well as gypsum, when the amount of water added with the anhydrite is equal to the amount that would have been present in the gypsum had gypsum been added instead of anhydrite.

This finding suggests that when gypsum is added and serves as a grinding aid, the water in the gypsum is released during grinding and essentially all of it becomes "free" water—as free as if it had been added separately from the calcium sulfate.

If this interpretation of what happens to the water content of the gypsum during grinding is correct, then it would seem to follow that, when gypsum is found in commercial cement, it would probably be more correct to regard it as a secondary rehydrated product than as original gypsum that had never been dehydrated. Following this line of reasoning, the finding of a mixture of gypsum and hemihydrate in a cement should probably be regarded as partial rehydration of dehydrated calcium sulfate rather than partial dehydration of gypsum.

It is suggested that a better understanding of factors involved in the phenomena concerning abnormal setting behavior of cements, as these are affected by the calcium sulfate in the cement, would be obtained if it were assumed that, regardless of the form or forms in which calcium sulfate existed when added to the grinding mill, when it comes out, it is probably essentially entirely in one or more essentially anhydrous states.

MR. PAUL SELIGMANN.[9]—It seems appropriate at this point to call attention to the excellent presentation and the ensuing discussions on false set in the *Proceedings of the Fourth International Symposium on the Chemistry of Cement.* Of particular interest in the present context is the discussion by Gaskin and McGowan, on pages 420–423 of that work, where an extensive experimental study of the factors producing false set is described. These workers find that the calcium sulfate present in cement has neither the X-ray diffraction pattern nor the properties to be expected of a mechanical mixture of calcium sulfate hemihydrate and gypsum. Rather, it consists of a mixture of thermally dehydrated and mechanically disorganized gypsum which is referred to as "cement gypsum."

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[7] Ideal Cement Co., Denver, Colo.

[8] Engineer, Concrete Division, U. S. Army Engineer Waterways Experiment Station, Jackson, Miss.

[9] Senior Research Physicist, Applied Research Section, Portland Cement Assn., Research and Development Division, Skokie, Ill.

Although the state of the calcium sulfate in cement is not well defined, nevertheless all the evidence, such as the analyses of aqueous extracts cited by Hansen in his symposium paper, points to the conclusion that the false set accompanies a very rapid precipitation of crystalline gypsum in the mix. It is granted that not all aspects of the mechanism of this stiffening are known. However, the phenomenon is not unique; similar stiffening occurs in other systems when a solid reaction product is rapidly precipitated in a paste with a high solids content. Gaskin and McGowan report that "As little as 0.5 per cent by weight of a well-nucleated dehydrated gypsum, when added to a pure silica-water paste, can produce severe stiffening if mixing is stopped at 2 min." In our work, we have found that such stiffening will occur when cement containing a small quantity of sucrose is mixed with a relatively low quantity of water. In this case X-ray studies show that a rapid precipitation of ettringite occurs at the time of stiffening.

With regard to the false set resulting from aeration of the cement, various workers have considered the effect to be due to precipitation of gypsum from a supersaturated solution resulting from inactivation of the C_3A surface by CO_2 but have been unable to confirm the presence of calcium carboaluminate hydrate. Gaskin and McGowan attributed the inactivation to a specific reaction between the gas and the solid surface different from the usual reactions between C_3A and carbonate *solutions* to yield the above-mentioned hydrate. In our own work, diffraction patterns have been made periodically of a sample of dry tricalcium aluminate packed into an X-ray sample holder and stored in a humid atmosphere. Although no rapid changes were observed, the material set to a hard mass after 60 days of exposure and X-ray diffraction revealed the presence of calcite and gibbsite rather than the carboaluminate. Thus the conclusion of Gaskin and McGowan was confirmed. Because of the short aeration time required for inactivation of the C_3A surface, it is clear that only a very slight reaction with CO_2 is sufficient for this inactivation.

MR. EMERY FARKAS.[10]—Test results obtained in our laboratory, at Dewey and Almy, substantiate Mr. Sawyer's finding regarding the limited grinding aid effect of water. In our test, clinker and anhydrite (to produce 2.1 per cent SO_3 in the cement) were interground at 210 F in the laboratory mill for 70 min. In subsequent grinds, increasing amounts of water were added to the mill with the clinker and anhydrite, and ground for 70 min. The results are shown in the following table:

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[10] Group Leader, Construction Materials Division, Dewey and Almy Chemical Division, W. R. Grace & Co., Cambridge, Mass.

Water Added, g	Blaine Surface Area, sq cm per g
0	3015
3.5	3300
10.0 (hemihydrate)	3575
33.0 (gypsum)	3725

In the third grind, the amount of water added was the same as if hemihydrate was used instead of anhydrite; and, in the fourth test, the same amount of water was added as if gypsum was used originally. Further tests, however, showed that beyond a certain point water does not increase the grinding effect and does not prevent ball coating.

CRYSTAL GROWTH AS A SOURCE OF EXPANSION IN PORTLAND-CEMENT CONCRETE*

By W. C. Hansen[1]

Synopsis

This paper reviews previous research on the development of pressure by crystals as they grow. The research shows that a salt crystal can grow under pressure if it is supported on a film of solution which can receive salt molecules from a solution that is supersaturated with respect to the crystal under pressure. It also shows that an ice crystal can grow under pressure if it is supported on a film of water in contact with a reservoir of water in which ice crystals cannot nucleate at the temperature at which the ice crystal is growing. The paper also outlines the conditions under which ice and salt crystals can grow and produce pressures in concrete and similiar porous materials.

Portland-cement pastes and gypsum plasters expand during setting and hardening, whereas they would be expected to contract because the volumes of the hydrated phases are less than the sum of the volumes of the water and reactants forming these phases. These expansions have been explained (**1**)[2] on the basis that the compounds under discussion can react directly as solids with water to form other solids. However, these expansions have also been attributed to crystals in contact creating pressure on each other by means of the process of growing. Recently, Jørgensen (**2**) and Ridge (**3**) suggested this as the mechanism involved in the expansion of gypsum plasters. This paper reviews and analyzes some of the literature on the growth of crystals in an effort to outline mechanisms whereby growing crystals may be sources of expansion in concrete and similiar products. However, no attempt will be made to analyze the extent to which such expansion may contribute either beneficially or deleteriously to the behavior of these products in service.

Development of Pressure by Salt Crystals

Becker and Day (**4**) appear to have been the first to demonstrate conclusively that a crystal can grow and lift an object when loaded with a weight. They placed a crystal on the bottom of a dish containing its saturated solution, covered it with a glass plate, and placed a weight on the glass plate. The crystal grew in height as the solution became supersaturated, either by cooling or by

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* Presented at the Sixty-sixth Annual Meeting of the Society, Atlantic City, N. J., June 23 - 28, 1963.

[1] Consulting Chemist, Valparaiso, Ind.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

evaporation. According to Taber (5), Knopp, in earlier work, did not obtain such growth; however, he had both weighted and unweighted crystals in the same dish. Taber (6), who undertook an extensive investigation of the behavior of crystals under different environments, verified the results of Becker and Day as well as those of Knopp. He found that a weighted crystal grew on all faces in a supersaturated solution but that the edges of the bottom face tended to grow faster than the other faces. These crystals tended to form a pyramid with stepped sides and a concave bottom. A section through such a crystal, in an exaggerated form, is depicted in Fig. 1.

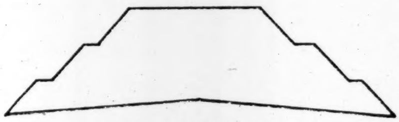

FIG. 1.—Diagram Depicting Section Through a Crystal Grown Under a Weight.

Taber explained the growth of such a crystal as follows: A crystal resting on the bottom of a dish is always separated from the floor of the dish by a film of solution, and the bottom face of the crystal is under the greatest pressure because it is supporting the weight of the crystal plus the applied load. The solution in contact with a crystal whose solubility increases with pressure has a maximum concentration under this face of the crystal. As the whole solution becomes more concentrated, either by cooling or evaporation, the denser portions settle to the bottom of the dish under the force of gravity. This raises the concentration of the solution at the bottom of the dish above that of a solution in equilibrium with the bottom face of the crystal. Solute molecules then tend to diffuse under the crystal through the film separating the crystal from the dish. However, as this occurs and the solution under the edges of the crystal becomes supersaturated with respect to those edges, the forces of crystallization draw the solute molecules onto the edges. This causes the edges to grow both horizontally and vertically. Very few solute molecules get beyond the edges. Accordingly the remainder of the bottom surface grows slowly with respect to the edges, and this surface gradually becomes concave. As the denser solution settles toward the bottom, the concentration of the solution increases with distance from the top surface. Hence the rates of growth of the uncovered faces increase with depth of immersion. This results in the formation of crystals with larger bases than tops as depicted in Fig. 1.

Taber also suspended crystals with pressure applied to two of the sides and observed their growth as the solution was concentrated. The crystals grew on all faces except those under pressure. They did not grow on these faces because no means was provided to maintain a supersaturated solution in contact with these faces as was done in the system of a crystal resting on the bottom of a dish under an applied load. In the latter, the dense solution could settle and provide supersaturation. Taber drew the following conclusions from his work (5):

1. A crystal will grow in a direction in which external forces oppose growth if the surface on which the forces are acting is in contact with a solution that is supersaturated with respect to it: and if the growing crystal is composed of a substance the solubility of which increases with pressure, then for any increase in the forces opposing growth a corresponding increase in the concentration of the solution is necessary.

2. If a crystal of such a substance is subjected to a greater pressure in one direction than in others (as for example when a weight is placed on a crystal) the surfaces that are under the lesser pressure will tend to limit the concentration of the solution and prevent

it from becoming supersaturated with respect to the surfaces that are rendered more soluble because of the greater pressure. In order that growth may also take place on the surface subjected to the greater pressure, some means must be provided for maintaining the supersaturation of the solution in contact with them.

Taber also studied the behavior of water and solutions in porous materials and found that solutions in subcapillary pores—pores in which the surface forces of the walls extend across the liquid between the walls—are not readily nucleated. Accordingly the solution in such pores will be supersaturated with respect to crystals growing in larger pores. Hence the crystals in the larger pores can receive material from the smaller pores. These crystals can then grow at this interface between liquid and the crystal and exert pressure on any material resting on the face of the crystal opposite to the growing face. This mechanism accounts for the columns of ice that grow up from the surface of soils under some conditions and for ice lenses formed beneath the surface of the soil.

Two conditions are thus seen under which growing crystals can exert pressure on materials opposing their growth: (1) the peculiar condition obtained when a crystal is weighted against the bottom of a container in a solution that can become supersaturated with respect to the bottom face of the crystal; (2) the condition in which a crystal in a pore is in contact with water or solution in a smaller pore. Taber suggested that the force responsible for the pressure developed by growing crystals is the force of crystallization which causes an expansion in volume when salts crystallize from solution.

It is desirable at this point to consider a system of a crystal resting on the floor of a container with another crystal resting on it under an applied load in a solution saturated with respect to the bottom crystal. The two crystals form an interface at which they are separated by a film of solution. The solution in this interface film is not in equilibrium with the solution as a whole because the pressure produced by the weight of the upper crystal and the applied load increases the solubilities of the crystals. Hence, salt molecules will diffuse from the solution in the interface into the main body of the solution. Since the portion of the solution adjacent to the edge of the interface will thus become more dense than the ambient solution, the more concentrated solution will flow downward toward the bottom of the container. Salt molecules in the more concentrated solution will be deposited on the faces of the lower crystal causing the lower crystal to grow at its sides as its upper surface dissolves and as the upper crystal disappears.

When a supersaturated solution is nucleated and crystals are formed, the crystals may be piled on each other. However, they will be separated from one another by a film of solution. Solute molecules will not diffuse into this film and attach themselves to surfaces in contact with one another so long as there are free surfaces to which they can become attached. If the solution is in equilibrium with these free surfaces and if the top crystals grow to sizes at which they exert significant pressure at the interface, the crystals will dissolve at this interface and grow at other surfaces. Thus, crystals growing in a solution that is being concentrated, or supersaturated by cooling, or into which salt molecules are diffusing, cannot exert any significant pressure on each other as long as the solute molecules can reach crystal faces that are not under pressure. If the process of supersaturation were continued, the mass of crystals would be converted into a single crystal without the development of pressure. Hence, theories that attribute expansions in concrete and similar products to crystals growing from solution against one another do not appear to be realistic. However, a solution that is saturated at one temperature, if cooled in a closed container, can exert pressure when solute crystals form because the system water-plus-crystals occupies a greater volume than that of the original solution. That is, the solute molecules expand in going out of solution to form the solid. Once the crystals have separated from the solution, any growth of the crystals by recrystallization will not change the volume of the system of crystals-plus-solution. The pressure exerted on the walls of the container is exerted through the solution phase and may be classified as hydraulic pressure.

DEVELOPMENT OF PRESSURE BY ICE CRYSTALS

The freezing of water in concrete may be a source of pressure from growing crystals. At the freezing point, water is similar in some respects to a saturated solution of a salt. Ice crystals and liquid water will coexist at this temperature just as salt crystals coexist with their saturated solutions. Raising the temperature of the water and of the solution will cause the crystals of ice and salt to disappear. Then if the temperatures are lowered to their original values ice and salt crystals may or may not reappear. In order for the crystals to reappear, it is necessary for crystal nuclei to form. For an understanding of nucleation in water, it is necessary to consider the structure of water.

The usual concept of the structure of water, that of a liquid consisting of single molecules closely packed and held together by weak van der Waals forces, does not explain many of the properties of water. Accordingly Bernal and Fowler (7) suggested a theory that proposed a structure similiar to that of many solids. For example, a block of quartz does not consist of individual or groups of polymerized molecules of SiO_2 but of Si^{4+} and O^{2-} ions arranged so that each Si^{4+} ion is surrounded by four O^{2-} ions. In other words, the block of quartz may be visualized as one large molecule of silica. Bernal and Fowler pictured a mass of water as being one molecule of water in which each O^{2-} ion is surrounded by four H^+ ions. This they believe gives three different intermolecular arrangements for water at different temperatures as follows: (1) an ice-like, tridymite-like form at temperatures below 4 C; (2) a quartz-like form between 4 and 20 C; and (3) an ammonia-like close-packed form at 200 to 240 C.

If this concept is accepted for water, a crystal nucleus will be formed when a group of water molecules changes from the quartz-like form to the tridymite-like form of ice. Chalmers (8) points out that, at the freezing point of 32 F, water is in equilibrium only with crystals of a certain size with flat surfaces. At this temperature, molecules leave the solid phase and enter the liquid phase at the same rate as molecules leave the liquid phase and become part of the crystal faces. The molecules leave the edges and corners easier than they do the flat surfaces and, accordingly, an irregularly shaped piece of ice becomes rounded. A very small crystal of ice will also melt and be deposited on the larger crystals. The criterion for size is the radius of curvature. If this is less than the critical radius, melting occurs; if larger, it does not. Hence, for an arrangement of molecules to serve as a nucleus for the crystallization of ice, at a given temperature, the aggregation must have a radius of curvature equal to or greater than some critical value.

According to Chalmers, at a temperature of 30 F, for example, the critical radius is about 3.2×10^{-6} cm. A sphere with this radius would consist of about 3 million molecules. The likelihood of this number of molecules of water assuming the correct structure is very remote. On the other hand, at −20 F the sphere of critical radius would contain about 250 molecules. Thus, a high degree of supercooling is required to nucleate pure water. However, foreign solid particles assist in the nucleation process; their effectiveness appears to depend upon the degree of matching of the structure of the foreign body with the structure of ice. In a porous material, such as concrete, the degree of supercooling depends upon the size of the pore containing the water. Ice cannot form in a pore of a given radius at a given temperature unless a crystal of a size that will not melt at that temperature can be formed in that pore. Ice will not form in small pores at temperatures at which it forms in larger pores, just as salts will not nucleate in very small pores.

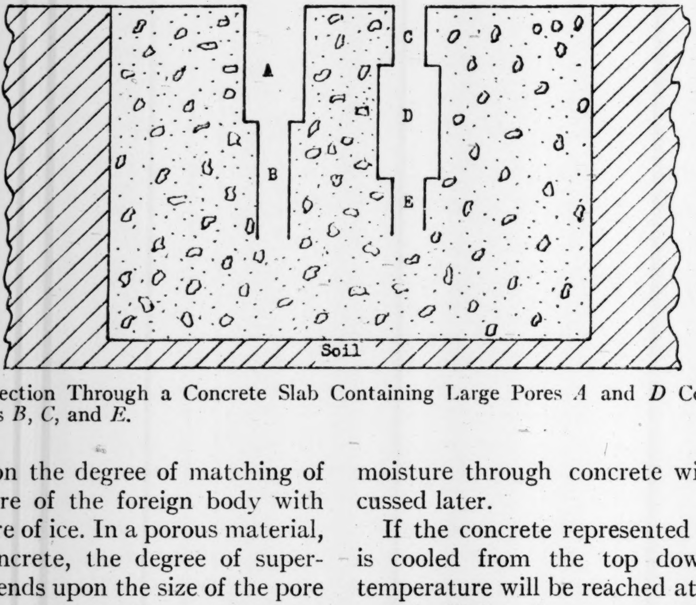

FIG. 2.—Section Through a Concrete Slab Containing Large Pores *A* and *D* Connected to Smaller Pores *B*, *C*, and *E*.

Figure 2 depicts a cross-section through two types of pores in a concrete slab resting on and surrounded, except for the top surface, by soil. In this paper the word pore is used to designate any space, regardless of size or shape, in a specimen of hardened cement paste which may be filled with water. Pore *A* (Fig. 2) is a relatively large pore that is open to the atmosphere through the surface of the concrete. Pore *D* is a pore of similar size located a short distance below the surface of the concrete which is open to the atmosphere through a much smaller pore *C*. Pores *B* and *E* are pores similar in size to the small pore *C*. These pores may, of course, be connected to others. The mechanism of the movement of moisture through concrete will be discussed later.

If the concrete represented by Fig. 2 is cooled from the top downward, a temperature will be reached at which ice nuclei will form in pores *A* and *D*. These nuclei will grow if the concrete is held at this temperature. As the crystals grow they will force water from pore *A* and from pore *D* through pore *C*, because ice cannot nucleate in *C* at the temperature at which it nucleates in *D*. Since water expands upon freezing, a point will be reached at which crystals of ice begin to exert pressure on each other in pores *A* and *D*. When this occurs, some crystals melt and the water molecules become attached to the faces of the largest crystals. This continues until the mass of crystals has been converted into one crystal that fills each pore, except for the films of water supporting the crystals on the floors of the pores.

This film in pore *A* is in contact with water in pore *B*, and the film in pore *D* is in contact with water in pore *E*. Taber (6) found that, under such conditions, water molecules from the smaller pores become attached to the bases of the crystals in the larger pores. In open pores, this causes ice to grow as a column above the surface. In soils in which the pores were covered with grains of soil, the growing ice columns exerted sufficient pressure to lift the particles covering them. If the covering is sufficiently heavy or strong enough to resist this pressure, the crystals cannot continue to grow because the melting point of the ice will be decreased by the pressure to the point at which the ice will be in equilibrium with the water in the smaller pore. This type of pressure, caused by growing crystals, can cause the concrete to spall off from over the pores or, if not relieved by spalling, can cause the concrete to expand and possibly to crack.

When ice crystals form in pores *A* and *D*, the expansion caused by the crystallization will tend to force water out of the pores. Since this pressure is developed in the presence of water, it is classed as hydraulic pressure. If this water cannot escape, either through pores out of the concrete or further into the concrete, it can cause the concrete to spall or to expand and crack just as growing crystals can.

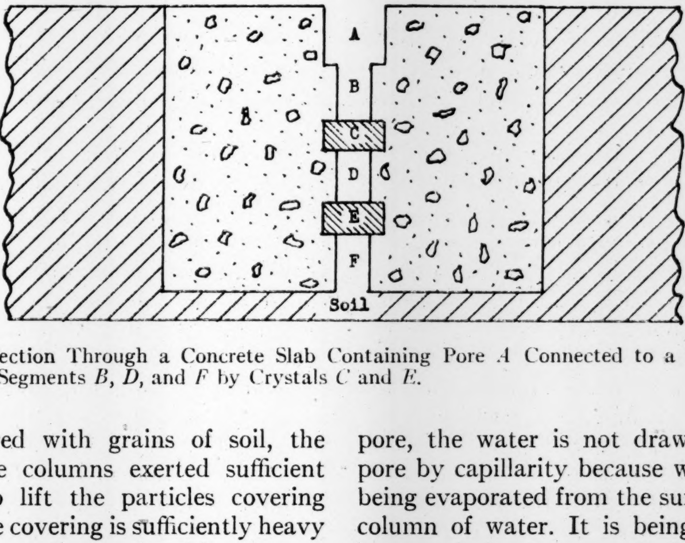

FIG. 3.—Section Through a Concrete Slab Containing Pore *A* Connected to a Smaller Pore Broken into Segments *B*, *D*, and *F* by Crystals *C* and *E*.

MIGRATION OF WATER AND SALTS IN POROUS SYSTEMS

When molecules of water from pores *D* and *E* (Fig. 2) become a part of the base of the ice crystal growing in the larger pore, the water is not drawn into the pore by capillarity because water is not being evaporated from the surface of the column of water. It is being drawn in because it loses energy in being transformed from liquid to solid and because of its tensile strength. By reference to the structure of water as proposed by Bernal and Fowler (7), it is seen that the breaking of a column of water involves the breaking of ionic bonds just as it does, for example, in the breaking of a crystal of quartz. A number of attempts have been made to determine the tensile strength of water. Briggs (9) attempted this by centrifuging boiled, distilled water in glass capillaries with internal diameters of 0.6 to 0.8 mm. The columns broke at negative pressure of 20 and 270 atmos, respectively, at slightly above zero and at about 10 C. However, there was no way of proving whether the columns broke by formation of air bubbles, or loss of adhesion to the glass, or because the tensile strength of the water was exceeded. Nevertheless, such measurements do show that water has a relatively high tensile strength and that water or a solution could exist in the pores of concrete under high tensile stresses.

The above explanation of the growth of ice crystals in concrete does not require the assumption that pore *A*, for example, is connected to the underlying soil by a continuous system of capillaries through which water is drawn to the pore. Figure 3 depicts pore *A* connected to the soil through smaller pores *B*, *D*, and *F* and crystals *C* and *E*. If the crystals are, for example, hydrated calcium sulfoaluminate, they will lose water as the water vapor pressure of their environment is decreased. Water of hydration will be removed from crystal *C* by the water in pore *B* as the water in this pore is put under greater tension by the growth of ice in pore *A*. However, as water molecules near the top surface of *C* are removed, they are replaced by water molecules from the remainder of the crystal. These, in turn, are replaced by molecules from the water in pore *D*. In this manner, hydrated crystals can perform as water-filled pores in transmitting water from the underlying soil to the surface of the concrete pavement. If the pores, either in the concrete or soil, are emptied of liquid water, molecules of water can migrate in the form of vapor by being adsorbed onto the surfaces of crystals *E* and *C*. They can then pass through the crystals and through pores *F*, *D*, and *B*, causing growth of the ice crystal in pore *A*; that is, an ice crystal can grow by being supplied with either liquid water or water vapor.

The lower surface of crystal *C* is no longer in equilibrium with the water in pore *D* as water migrates from this surface toward pore *B*. Accordingly, water molecules from pore *D* diffuse into crystal *C* as others leave crystal *C* to be drawn into pore *B* and to the ice crystal in pore *A*. Likewise, water is drawn from the soil through crystal *E* and pore *F* as the tension is increased in the water in pore *D*.

The above mechanism for the migration of water can be applied to the migration of salts in concrete. Suppose, for example, that the soil of Fig. 3 contains gypsum, and the solution in pore *F* is saturated with respect to gypsum; the sulfoaluminate crystal *E* is then in contact with a saturated solution of gypsum. Calcium sulfoaluminate ($3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot 31H_2O$) dissolves in water to give a solid phase that contains slightly less $CaSO_4$ than required by the above formula. This, of course, is not the case when the sulfoaluminate crystal is in contact with a solution containing a relatively large number of calcium and sulfate ions, such as a saturated solution of gypsum. Therefore, as the face of crystal *F* in contact with the solution in pore *D* loses some calcium sulfate to this solution, that face contains less calcium sulfate than does the face in contact with the solution in pore *F*. When this occurs, calcium and sulfate ions diffuse through crystal *E* into the solution of pore *D* and upset the equilibrium of this solution with crystal *C*. Calcium and sulfate ions then diffuse through *C* into pores *B* and *A*. Evaporation of water from pore *A* can nucleate the gypsum in the solution in this pore and set up a condition whereby molecules of calcium sulfate can continue to migrate from the soil into pore *A* by the process described below.

Probably most concrete technologists have observed salt crystals growing from some concrete structures and in some cases from plastered walls. Generally these are observed in locations in which the structure is in contact with wet soil or with water carrying salts. Griffin and Henry (10) show some good pictures of sodium-chloride whisker crystals growing from what they class as wet cups. In such a cup, a specimen of concrete, to which sodium chloride had been added in the mixing water, was exposed to the vapor of distilled water. The assembly was then stored in an atmosphere of fixed temperature and relative humidity. In this system, the under side of the concrete specimen was in contact with air at approximately 100 per cent relative humidity, and the upper side was in contact with air at a lower relative humidity. The following observations of Griffin and Henry regarding the growth of sodium chloride crystals are readily explained by the results of Taber's work (5):

> The rate of growth appeared to be somewhat more rapid in the 20 per cent relative humidity than in the 50 per cent relative humidity; however, the ultimate amount of growth over 3 to 4 months, in general, appeared to be about the same for most specimens....
>
> On some specimens the crystals were like lamb's wool and on others they were stubby and short.
>
> Individual crystals are striated and have the appearance of being extruded from a die. They definitely appear to have grown from the base. In fact, if viewed at the proper time under a low-powered microscope, the whiskers may be observed at the break through a crust in the matrix that has already been lifted above its original position by the formation of the whisker.

In discussing observations made in a building built with concrete containing sodium chloride the authors state: "These whiskers emerged through a layer of paint and caused the surface of the walls to flake off to a depth of $\frac{1}{32}$ in."

These observations by Griffin and Henry are readily explained by reference to Fig. 2. Suppose that the concrete depicted in Fig. 2 is saturated with water and that the soil is wet. Suppose, also, that the pores of the concrete and soil contain a solution of sodium chloride that can be caused to nucleate with sodium chloride crystals, either by evaporation of water or by cooling the solution. However, the solution in pores *B*, *C*, and *E* will not nucleate at the temperature at which the solution nucleates in pores *A* and *D*. Hence, when nucleation occurs in pores *A* and *D* some of the salt crystallizes. The solution in these pores is then not in equilibrium with the solution in pores *B*, *C*, and *E*. Accordingly salt molecules will diffuse from the solution in these pores into the solution in the larger pores. This reduces the concentrations in pores *B*, *C*, and *E* below that of the solution in the other pores. A system is thus set up in which salt molecules are migrating from the interior of the concrete and from the soil into pores *A* and *D* through pores *B* and *E*.

Now suppose that once nucleation occurs, the temperature remains constant and evaporation of water from pores *A* and *D* ceases. As long as the temperature is maintained constant, the solution in pores *A* and *D* will remain unsaturated with respect to the solution in pores *B* and *E* provided that the solution in these pores can draw salt from the concrete and soil. Accordingly salt molecules will diffuse into pores *A* and *D*, causing the crystals in them to grow. As previously pointed out, this crystallization of salt is an expansion reaction, and this will force solution to overflow from pores *A* and *D* and spread over the surface of the concrete where it can form a salt crust. As crystals in pores *A* and *D* grow, they tend to fill all available space and exert pressure upon one another. However, as previously discussed, crystals under pressure will dissolve. This will cause the formation of a single crystal in pore *A* and in pore *D*. The crystal in pore *A*, for example, which completely fills the pore, is supported on the floor of the pore by a film of solution which is in contact but not in equilibrium with the solution in the smaller pores, *B* and *E*. Salt molecules from pore *B* diffuse into this film and through the forces of crystallization become a part of the base of the crystal in pore *A*. This lifts the crystal above the surface of the concrete and forms a whisker or a column depending on the diameter of pore *A*. The growing column will lift any paint or crust that covers pore *A*, and it may grow for some distance above the surface of the concrete. In the case of pore *D*, the growing crystal will exert pressure on the concrete covering the pore. If the crystal can develop sufficient pressure, the layer of concrete will spall off. Lesser pressure might cause some expansion of the concrete.

In this process, in which salt molecules diffuse from smaller into larger pores, the relative humidity of the atmosphere plays no part, because, once the pore is filled with a single crystal, there is no evaporation from that pore. This explains why Griffin and Henry found that the ultimate amount of growth was independent of the humidity of the atmosphere and that the crystals grow from the base and are extruded through the pore as through a die.

SCALING OF CONCRETE BY GROWING CRYSTALS

Concrete technologists are familiar with two types of scaling:

1. Scaling of piles, piers, and so on, where the structures are partially immersed in salt-bearing water or in salt-bearing soil. This type of scaling is generally confined to a small area immediately above the water or soil surface.

2. Scaling from the surface of a structure such as a pavement slab, sidewalk, or floor where the slab is embedded, except for its surface, in salt-bearing soil, or where salts or solution come in contact with the surface. These may be applied to remove ice, or they may fall on the concrete through operations carried out on the floor or pavement. An example is the deicing of airplanes with solutions of organic compounds such as glycerin and glycols.

In the first case, salt-bearing water diffuses into pores of the drying concrete whenever water evaporates from the concrete extending above the water or soil. As evaporation proceeds, a point can be reached at which salts begin to crystallize in a pore, such as *D* of Fig. 2, which is slightly below the surface of the concrete. Once crystals form, the solution in the pore will draw salt molecules from the solutions in smaller pores. Eventually the pore becomes filled with a single crystal. Further growth of the crystal may cause spalling of concrete from above that pore. Spalling, of course, will expose other pores to the atmosphere, and the scaling can continue to greater and greater depths. Also, the pore becomes an open pore, once the covering layer has spalled off, and then the crystal may grow as a whisker or column above the surface.

In the second case, a solution of salts covers the surface of the slab when ice is melted with a salt. Salt molecules from this solution tend to migrate into the concrete either by diffusion in the water in pores or by diffusion through other crystals. Salt molecules can accordingly enter concrete that is saturated with water and which does not imbibe additional water. If the surface of the slab is then sealed by a layer of ice, further cooling will tend to cause crystals of salt to form in the pores. Since the separation of salt from the solution is an expansion reaction, and since the solution cannot escape out through the surface of the slab, the formation of crystals will force solution into pores that are not already filled. Thus, each freezing drives salt-bearing solution deeper and deeper into the concrete. Salt-bearing solution will also be driven deeper into the concrete if the water freezes in a pore near the surface, because the separation of ice crystals is an expansion reaction. This process of melting ice with salt and subsequent freezing of water causes the liquid phase of the concrete to become a solution of the deicing salt.

Either cooling or evaporation can start crystallization of salt in some of the pores of this salt-bearing concrete. This will set up a condition under which salt molecules can be drawn into a pore, such as *D*, for example, and under which the salt crystals can grow into a single crystal. This crystal can continue to grow and exert pressure on the concrete covering the pore and, of course, may cause scaling if the concrete cannot resist this pressure. If the concrete can resist this pressure without scaling, it may expand and crack.

There are thus two conditions under which a crystal can grow and exert pressure on materials opposing its growth. One is the condition in which a weighted crystal rests on the floor of a container on a film of solution that is supersaturated with respect to the bottom face of the crystal. The other is the condition in which a single crystal fills a container that opens into another container filled with a solution supersaturated with respect to the film of solution supporting the crystal. Actually the two conditions are identical in that solute molecules from the surrounding solution, in the first case, and from the underlying solution, in the second case, are diffusing into a film of solution that is supporting a single crystal on the floor of the container. The molecules, through the forces of crystallization, lift the crystal against its own weight or any force acting on the surface of the crystal to hold it against the floor of the container. Salt, but not ice, crystals can grow and exert pressure under the first condition, whereas, under the second condition, either ice or salt crystals can grow and exert pressure against a force acting on the face opposite the growing face. Ice crystals cannot produce pressure under the first condition because there is no way of producing in a container of water the equivalent of the supersaturated solution at the bottom of the container that can be obtained in a solution where a denser solution can settle under the force of gravity.

Scaling of concrete has been observed when solutions of organic liquids, such as glycerin and glycols, are used to deice airplanes on concrete storage areas. These materials act like acids in being able to dissolve calcium hydroxide and other calcium compounds. They accordingly exert a corrosive action on concrete. They also greatly increase the amount of calcium hydroxide that can be dissolved by water. For example, Linke (11) gives the solubility of calcium hydroxide in water as 0.159 g per 100 ml of solution; 0.301 when the solution contains 7.15 per cent of glycerin, and 3.55 when the solution contains 69.2 per cent glycerin by weight. The solid phase that separates first from these solutions is calcium hydroxide. The organic liquids can diffuse into the concrete by the mechanism described above for the diffusion of salt molecules. The organic compounds as they diffuse into the concrete tend to increase the concentration of calcium hydroxide in the liquid in the pores. Also, when water evaporates from this solution, concentration of the organic component increases the concentration of calcium hydroxide in the solution. If this solution is diluted by a rain, for example, some of the calcium hydroxide crystallizes from the solution because dilution decreases the solubility of the calcium hydroxide. Such crystallization, for example, in pore *D* of Fig. 2, unsaturates the solution with respect to the solution in pore *E*. Hence, calcium hydroxide molecules can diffuse from pore *E* into *D*, and the crystals in pore *D* can grow into a single crystal. This crystal can then grow from the base and break the concrete from above pore *D*. If the concrete above *D* could withstand the pressure, the crystal in the pore could cause the concrete to expand and possibly to crack. It appears, therefore, that organic liquids that have the property of forming solutions in which the solubility of calcium hydroxide increases with increasing concentration of the organic liquid can give rise to pressures in concrete by growing crystals.

DEVELOPMENT OF PRESSURE BY CHANGES IN TEMPERATURE

Helmuth (12) observed a delayed expansion when a specimen of hardened cement paste, stored for 3.5 yr in a saturated solution of calcium hydroxide, was cooled rather rapidly from 25 to 14 C and then allowed to obtain volume constancy by standing at this temperature for 75 min. The specimen contracted approximately 0.02 per cent during cooling, and this decreased to approximately 0.016 per cent while the specimen was held at 14 C. Delayed contractions of similiar magnitude were observed when specimens were heated and allowed to obtain volume constancy at the higher temperature. The delayed expansion was explained on the basis of a delayed diffusion of water from capillary pores into gel pores. The delayed contractions were explained on the basis of the reverse flow of water, that is, from gel pore into capillary pore.

Helmuth follows the classification of pores proposed by Powers (13) in which a gel pore is so small that there is space for only a few layers of adsorbed water on the solid particles enclosing the pore. Capillary pores are large enough to contain unadsorbed or free water. One might picture a capillary pore in a water-saturated hardened cement paste as being a system of water-containing space enclosed by walls of colloidal-size bricks. Each brick can be visualized as being one of the reaction products of cement plus water, with surfaces made up of a few layers of adsorbed water. Each brick is in contact with others. The contacting layers of adsorbed water constitute gel pores. It is of interest to examine this system to determine whether the delayed volume changes observed by Helmuth are associated with changes in the sizes of crystals, that is, the sizes of the colloidal bricks forming the walls of the capillary pores.

At equilibrium, at a given temperature, the solid walls of this system are in equilibrium chemically and physically with the liquid phase in the capillary pores. From a kinetic viewpoint, solute and water molecules are leaving the solid phase to become a part of the liquid phase at the same rates as solute, and water molecules are leaving the liquid phase to become a part of the solid phase. The specimen used by Helmuth was made from a low-alkali cement and was stored for 3.5 yr in a saturated solution of calcium hydroxide. The liquid phase of this specimen would be about as dilute as possible for a solution in equilibrium with a hardened portland-cement paste because of the low alkali content of the cement and loss of alkalies to the water during storage. The solid phases of the pore walls, neglecting the iron phases, very likely consisted of compounds with compositions close to the following: $3CaO \cdot 2SiO_2 \cdot 3H_2O$; $4CaO \cdot Al_2O_3 \cdot 19H_2O$; $3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot 31H_2O$; and $Ca(OH)_2$. Since the solution was as dilute as possible, the adsorbed water content of the solid phase should have been the maximum possible in a hardened cement paste. That is, the gel pores, as defined above, should have been as large as it is possible to obtain in a hardened portland-cement paste. The liquid phase of a hardened cement paste contains only minor amounts of the component oxides of the solids other than CaO, SO_3, and the alkalies. Hence, one probably can safely consider the liquid phase of the Helmuth specimen as being a dilute solution of calcium hydroxide and calcium sulfate.

Brunauer (14) shows three structures for $3CaO \cdot 2SiO_2 \cdot 3H_2O$, which he calls tobermorite (G), to illustrate the manner in which adsorbed and chemically combined water (water of hydration) might be distributed in the crystals of this solid. One structure shows the water held between layers of sheet-like crystals, and the others depict two ways in which water can be adsorbed on fibers produced by rolling up the sheets. Brunauer believes that the crystals of tobermorite (G) containing three molecules of water, as might be the composition of the compound in the specimen under discussion, contain one mole of water between layers in the sheets. This layer of water (water of hydration) is lost progressively as the vapor pressure of the environment is reduced. In a dried specimen, this layer of water is regained progressively as the vapor pressure of the environment is increased. Brunauer also believes that, when tobermorite is dried in an atmosphere with a vapor pressure of 8×10^{-3} mm Hg and a water content of 2.8 moles, 0.3 moles of the water is in the form of adsorbed water. Therefore, the water in these compounds that can be classed as chemically combined water of hydration as well as the adsorbed water can be lost progressively as the vapor pressure of the environment is reduced. The reverse is that both chemically combined water and water of adsorption can be gained progressively as the vapor pressure of the environment is increased. It seems evident from this that it is extemely difficult, if not impossible, to distinguish clearly between water of hydration and adsorbed water in colloidal-size crystals. In such crystals the adsorbed water may constitute an appreciable percentage of the total water content of the crystal. This progressive gain and loss of water with changes in the ambient vapor pressure applies not only to the adsorbed water and water of hydration of tobermorite (G) but also to these classes of water in the other solid phases of the hardened cement paste.

A convenient way of deciding what would occur in the system, capillary pore consisting of walls of colloidal materials and a saturated solution, when the temperature is changed is to consider what effect the change would have on the vapor pressure of a solution such as that in the pore. When the temperature of the specimen under discussion is lowered, the vapor pressure of the solution would decrease for two reasons. If there were no change in the solute concentration of the solution, the vapor pressure of the solution would behave like that of pure water and decrease with the decrease in temperature. However, the solubility of calcium hydroxide increases with decrease in temperature- Hence, since the solution is in contact with solid calcium hydroxide, the concentration of the solution would increase with the decrease in temperature. Both of these effects tend to lower the vapor pressure of the solution in the capillary pore, causing the solids of the walls to lose water to the solution. That is, the crystals should decrease in size. On the other hand, an increase in temperature should raise the vapor pressure of the solution and cause water to be lost from the solution to the solids. This increase in temperature will cause the crystals to grow.

Since water occupies more volume as a component of the solution than it does as a component of the solid phase, that is, as either adsorbed water or water of hydration, loss of water to the solution should cause expansion. This loss of water, if it lagged behind the rate of thermal shrinkage, could explain the delayed expansion observed by Helmuth. This conclusion is in conflict with that of Helmuth in that he concluded that the loss of water from the solution to the solid, that is, from capillary pore to gel pore, caused the expansion. However, it seems impossible to postulate that liquid water is moving, under a change in temperature, from a capillary pore into a gel pore and remaining as liquid water. If it moved as a liquid from the capillary pore into the gel pore and there became a part of the solid, the system would have to shrink.

It might be argued that the loss of water from solid to solution would not expand the specimen because the specimen is permeable to water. However, it must be realized that this flow of water from solid to solution is occurring simultaneously in all pores. Therefore, the pores near the surface of the specimen must lose their excess water to the ambient atmosphere before the underlying pores can lose water to the near-surface pores. It seems reasonable to postulate that this diffusion of water would be slow enough to permit transient expansions in a saturated specimen treated in a manner that permitted flow of water from the solids to the solution phase.

The above discussion has been concerned with the solution phase of a low-alkali cement paste. Some cements contain up to about 1 per cent of alkalies. The liquid phase of such a cement paste probably is a fairly concentrated solution of the alkali hydroxides. Cooling of such a solution might cause the precipitation of some of the hydroxides because their solubilities decrease with a decrease in temperature. This would raise the vapor pressure of the solution and cause water to flow from the solution to the solid phase. This would cause the specimen to contract. However, if this specimen is immersed in relatively pure water, alkalies would be leached from the specimen. This would raise the vapor pressure of the solution in the capillary pores. The water content of the solid phase might then increase. Since the specimen is immersed in water, the water taken up by the solid could be replaced in the solution of the pore by the immersion water. This then could cause expansion, and this would be a case of the crystals growing by increasing either their adsorbed water, their chemically combined water, or both.

If the partially dried paste is exposed only to water vapor and the water content of the vapor is increased, some of the solid phases can increase their chemically combined and adsorbed water contents. In this way, crystals can grow without the presence of liquid water. This growth can cause expansion.

SUMMARY

There are four ways in which crystals can grow and produce pressure in concrete:

1. Salt crystals existing in a pore take up solute ions from a solution with which they are in contact but which cannot nucleate at the ambient temperature because of the size of the container.

2. Ice crystals existing in a pore take up molecules of water from water with which they are in contact but which cannot nucleate at the ambient temperature because of the size of the container.

3. Colloidal crystals may increase their adsorbed and chemically combined water contents when they enclose a capillary pore filled with a solution if the vapor pressure of the solution is increased and if the water removed from the solution in the pore can be replaced by water from an outside source.

4. Colloidal crystals, in the absence of liquid water, may increase their adsorbed and chemically combined water contents if the water vapor pressure of their ambient atmosphere is increased.

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VOLUME CHANGE AS A MEASURE OF FREEZING-AND-THAWING RESISTANCE OF CONCRETE MADE WITH DIFFERENT AGGREGATES*

By Milton H. Wills, Jr.,[1] Henry A. Lepper, Jr.,[2] R. D. Gaynor,[1] and Stanton Walker[1]

Synopsis

A freezing-and-thawing procedure, based on the method proposed by Powers in 1955 (**1,2**)[3], appears to offer a simplified approach to studies of concrete durability. The test involves relatively long periods of immersion of concrete test specimens in water, together with a few intervening cycles of freezing and thawing. The method has not been nationally standardized nor widely used. The investigation described here was made with the objective of learning more about its applicability and significance. Concrete with two different coarse aggregates and curing conditions were tested by the approach just described and by ASTM Method C 291.[4] There was good correlation between the two test methods. The results offer promise that the simplified approach, with procedures standardized and refined, may provide a good tool for investigating concrete durability.

Tests of the resistance of concrete to freezing and thawing appear to be the best approach to studies of the durability of concrete exposed to weathering. Many different procedures have been used and ASTM has standardized four methods[4] which incorporate the principal features of those most used and most promising. These test methods provide information helpful in judging concrete durability, but their correlation with the effects of aggressive weathering conditions leaves much to be desired. Further, the tests are time-consuming and laborious. The need for further studies of test procedures is evident.

What appears to be a simplified approach, involving relatively long periods of immersion of concrete test specimens in water, together with a few intervening cycles of freezing and thawing, has been suggested by Powers (**1,2**).[3] The principal measure of specimen durability is the period of immersion required to saturate the concrete to the point that a single freezing-and-thawing cycle will produce significant "dilation," which is defined as expansion resulting from the formation of ice that may exceed the contraction resulting from cooling.

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper.

[4] Tentative Method of Test for Resistance of Concrete Specimens to Rapid Freezing and Thawing in Water (C 290), Tentative Method of Test for Resistance of Concrete Specimens to Rapid Freezing in Air and Thawing in Water (C 291), Tentative Method of Test for Resistance of Concrete Specimens to Slow Freezing and Thawing in Water or Brine (C 292), and Tentative Method of Test for Resistance of Concrete Specimens to Slow Freezing in Air and Thawing in Water (C 310), 1961 Book of ASTM Standards, Part 4.

The Powers test procedure offers a unique opportunity to study the effects of curing and moisture condition on the resistance to freezing and thawing. It provides procedure for a more direct evaluation of moisture content, less complicated by the simultaneous effects of freezing-and-thawing cycles than do standard procedures.

In view of these considerations, an investigation of certain features of the "Powers" method was carried out in the Graduate School of the University of Maryland. Comparisons were made with the results of the ASTM standard procedure, C 291.[4] The work was done in the Department of Civil Engineering through the Research Fellowship Foundation, sponsored jointly by the National Sand and Gravel Assn. and the National Ready Mixed Concrete Assn. Milton H. Wills, Jr., was the research fellow, and this paper is based upon the thesis which he prepared in partial fulfillment of the requirements for a Master of Science degree.

DESCRIPTION OF THE INVESTIGATION

The investigation consisted of measuring the resistance to freezing and thawing of concretes made with two different aggregates for two curing conditions: (1) continuously wet and (2) wet followed by a period of drying. The two freezing-and-thawing methods were an adaptation of the Powers procedure, described in detail later and hereafter referred to as the "slow cycle," and the ASTM Designation C 291, hereafter called the "rapid cycle."

TABLE 1.—AGGREGATE CHARACTERISTICS.

Coarse aggregates were separated into individual sizes and recombined to contain 25 per cent each of 1 to ¾ in., ¾ to ½ in., ½ to ⅜ in., and ⅜ in. to No. 4.

Aggregate	Specific Gravity (Saturated Surface — Dry)		Absorption, per cent		Voids (Dry Rodded), per cent	Fineness Modulus	Particles Lighter than 2.40 g, per cent
	24-hr Soak	Vacuum Saturated	24-hr Soak	Vacuum Saturated			
Coarse:							
Limestone	2.62	2.62	3.5	3.5	40.1	6.90	3
Chert	2.37	2.41	4.3	6.2	35.5	6.90	40
Fine:							
Quartz	2.62	...	1.2	...	...	2.52	0

Materials:

The coarse aggregates were a crushed limestone and a chert gravel (see Table 1). These were selected because of their widely different physical and chemical characteristics and because of their known poor resistance to freezing and thawing in concrete in a saturated condition. A further consideration was the considerable amount of data available from their use in earlier freezing-and-thawing studies.

The limestone was an impure dolomitic material with which the Iowa State Highway Dept. had unsatisfactory experiences (3). It was also used in the cooperative freezing-and-thawing tests sponsored by the Highway Research

Board (see Appendix D of reference (**4**)). The chert is typical of many gravels occurring in Tennessee, Missouri, and other mid- and south-central states. Such materials have been covered in several investigations of which those listed among the references are representative (**5**–**8**).

The coarse aggregate was well graded from No. 4 to 1 in., and the same high-quality quartz sand was used as fine aggregate in each case (see Table 1). The coarse aggregates were saturated under vacuum and soaked 24 hr prior to incorporation in concrete. Saturation was accomplished by oven-drying the aggregates, subjecting them to a vacuum of less than 5 mm of mercury for ½ hr, and breaking the vacuum with water. The sand was moistened the day preceding its use and was covered to prevent evaporation. A blend of equal amounts of five brands of type I cement, purchased on the local market, was used. A neutralized Vinsol resin solution added with the mixing water was used as the air-entraining agent.

Concrete:

Concrete was proportioned by conventional procedures to contain 5.5 sacks of cement per cubic yard, and to have an air content of 4 to 5 per cent and a slump of 3 to 4 in. (see Table 2). It was mixed in 2.2-cu ft batches in a 3½-cu ft tilting mixer. Two batches were mixed on different days for each coarse aggregate. Eighteen 3- by 4- by 16-in. prisms were molded from each batch. Because of limitations on freezing capacity, the two "rounds" were mixed about two months apart.

The two 3- by 16-in. faces of each prism were equipped with brass gage plugs on 10-in. centers for routine length measurements with a Whittemore strain gage. Prisms for exposure to the slow cycle had additional steel gage plugs projecting from each end along the longitudinal axis. Thermocouples were installed at the geometric center of each prism. To avoid damaging the gage plugs and thermocouples, the concrete was compacted by vibrating the molds with a small, high-frequency air vibrator.

The two curing procedures, for the principal freezing-and-thawing tests, were (1) one day in moist room (in

TABLE 2.—CHARACTERISTICS OF CONCRETE.

Coarse Aggregate	Round	b/bo[a]	Sand, per cent total aggregate	Cement, sack per cu yd	Mixing Water, gal per cu yd	Slump, in.	Air Content,[b] per cent
Limestone	A	0.70	39.2	5.54	34.4	2.7	4.4
	B	0.70	39.2	5.55	35.0	3.0	4.1
	avg	0.70	39.2	5.55	34.7	2.9	4.3
Chert	A	0.70	35.2	5.55	32.7	3.4	4.6
	B	0.69	35.2	5.50	33.9	4.0	4.7
	avg	0.70	35.2	5.53	33.3	3.7	4.7

[a] b = Solid volume of coarse aggregate per unit volume of concrete;
bo = Solid volume of coarse aggregate per unit volume of dry rodded coarse aggregate.
[b] Determined gravimetrically in accordance with ASTM Method C 138 (see footnote 5).

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[5] Method of Test for Weight per Cubic Foot, Yield, and Air Content (Gravimetric) of Concrete (C 138), 1961 Book of ASTM Standards, Part 4.

molds), followed by 13 days in saturated limewater (wet curing) and (2) the same as the preceding followed by seven days in air having a relative humidity of 40 to 60 per cent (dry curing). All of the curing operations were at temperatures controlled within ±1 F and within the range of 70 to 75 F.

Sixteen of the eighteen prisms cast from each batch (64 in all) were used for the principal freezing-and-thawing tests. Eight from each batch were cured "wet" and eight "dry." From each of these groups, six prisms were subjected to the slow cycle and two to the rapid cycle. The two remaining prisms from each batch were used in tests not reported here.

Slow Cycle:

The slow cycle consisted of an initial cooling of the specimens to 0 F, followed by immersion in water at 40 F for two weeks, when another slow cycle was performed. This cycle of slow freezing followed by immersion for two weeks was repeated until the relative modulus of elasticity (relative *E*) was reduced 50 per cent or until the specimens were transferred to the rapid cycle for further tests.

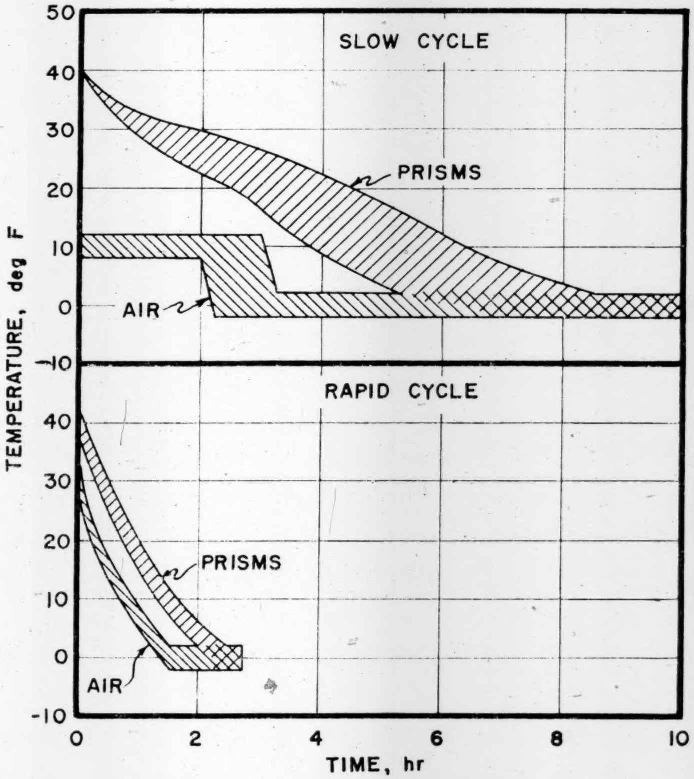

Fig. 1.—Cooling Rates for Freezing Tests.

Before freezing, the prisms were measured for length, weight, and fundamental frequency. They were then sealed in polyethylene bags to minimize drying during freezing and were placed on end in a vertical comparator in the freezing

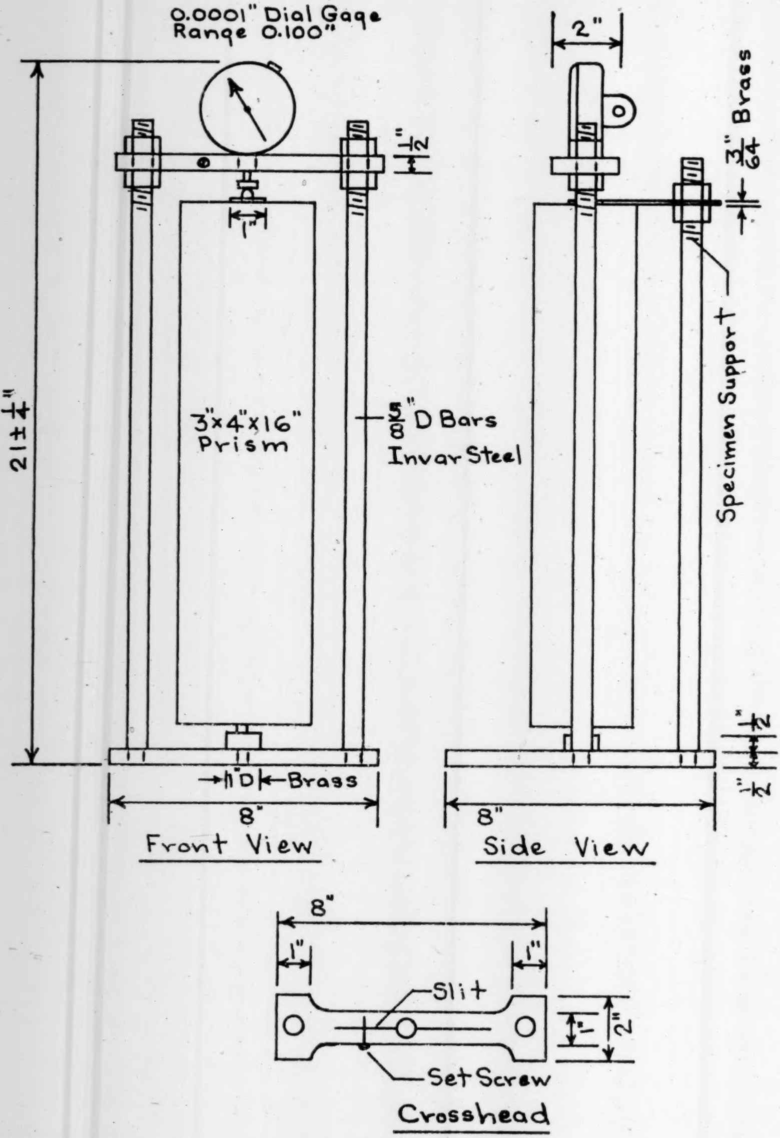
0.0001" Dial Gage
Range 0.100"
21 ± 1/4"
1/2"
1"
3"×4"×16"
Prism
5/8" D Bars
Invar Steel
1" D
Brass
8"
Front View
2"
3/64" Brass
Specimen Support
1/2"
1/2"
8"
Side View
8"
1"
1"
Slit
1"
2"
Set Screw
Crosshead

FIG. 2.—Vertical Comparator.

chamber. The ambient air temperature was controlled at 10 F for 2 to 3 hr, depending on the rate of cooling, and then at 0 F to reduce the specimen temperature from 40 F to 0 F in a period of about 8 hr.

The range in air and specimen temperature is shown in the upper portion of Fig. 1. Highly saturated concrete had the slowest cooling rate while dry concrete had the fastest. Most of the concrete cooled at rates near the average of these two extremes. Data for "cooling curves" were secured from simultaneous measurements of specimen length and temperature during the cooling cycle. When specimens reached 0 F, they were removed from the comparators and placed in 40-F water. On the following morning, they were again measured for length, weight, and relative dynamic modulus and returned to water at 40 F for 13 days.

The vertical comparator is shown in Fig. 2. Six of these were built, permitting simultaneous measurements of the six specimens at one time. Each comparator consisted of a dial gage with 0.0001-in. divisions mounted on a stand designed to hold the specimen vertical along its longitudinal axis. The parts of the comparator were made of selected lengths of brass, invar steel, and mild steel in an attempt to eliminate the effects of temperature on the comparator. This was accomplished to the extent that the effects on the shape of the cooling curves were negligible, as was verified by comparisons made with an invar steel bar.

Five measurements related to specimen condition were derived from the slow cycle. These were dilation, residual length change, cumulative length change, moisture content, and modulus of elasticity measured dynamically. The first three require definition and some discussion of the cooling cycle may be helpful.

Concrete, of course, contracts as it is cooled. When highly saturated concrete is cooled to low temperatures, however, the formation of ice results in expansion which may exceed the contraction due to cooling (9–12). This expansion has been called dilation (1,2). Figure 3 illustrates the method by which it was measured in this investigation, that is, at the point of maximum specimen length which occurred after freezing had started. By this method dilation less than 15 μin. per in. was difficult to detect. See the curves for the second and fourth cycles in the righthand portion of Fig. 4.

Residual and cumulative length change were determined with the Whittemore strain gage. The residual length change, for any cycle, is the difference in lengths measured 12 to 14 hr before placing the specimen in the freezing chamber and 12 to 14 hr after thawing.

It may be considered as somewhat akin to permanent set resulting from a single slow cycle. In both instances the specimen would be at equilibrium with the 40-F thawing water. The cumulative length change at any time is the difference in length from that measured at the beginning of the test. Moisture content was determined by drying the specimens to constant weight at 230 F at the end of the test.

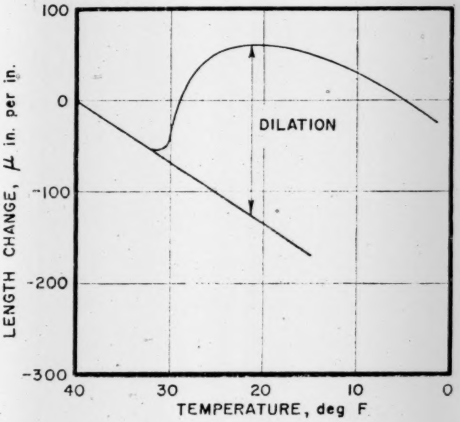

Fig. 3.—Method of Measuring Dilation.

The "dynamic modulus" was measured in accordance with ASTM Method C 215.[6]

Rapid Cycle:

The conventional rapid cycle was made in accordance with ASTM Method

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[6] Method of Test for Fundamental Transverse, Longitudinal, and Torsional Frequencies of Concrete Specimens (C 215), 1961 Book of ASTM Standards, Part 4.

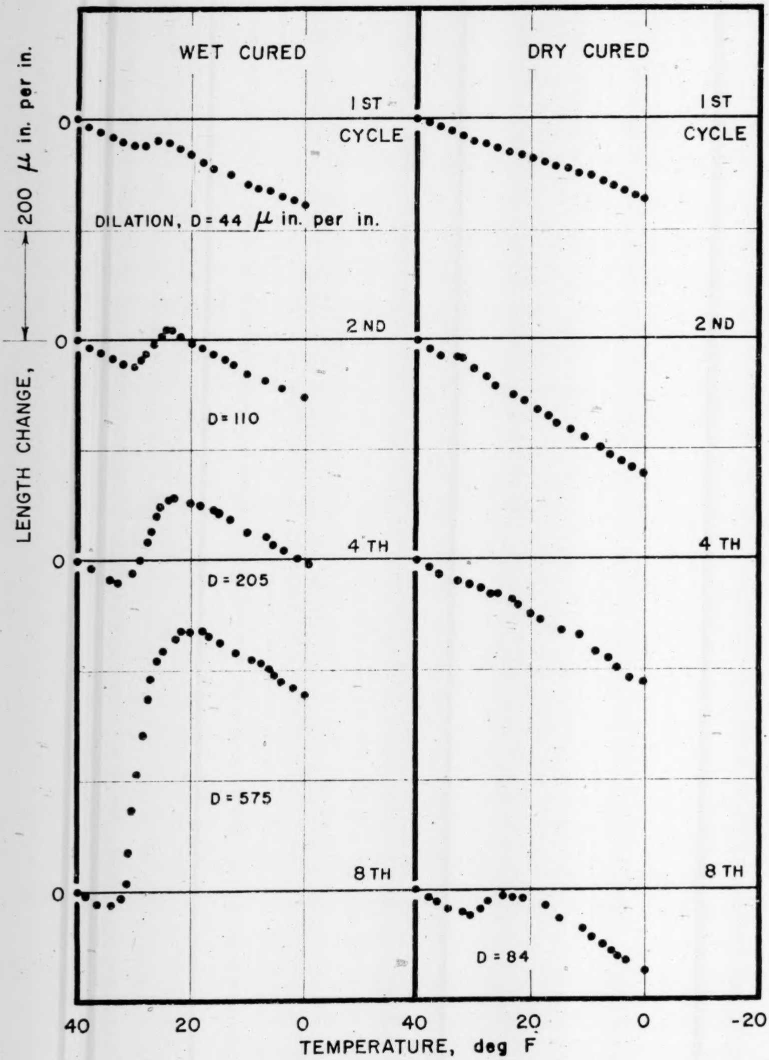

FIG. 4.—Typical Cooling Curves for Limestone Concrete in the Slow Cycle.

C 291. Specimens were frozen in air at 0 F for 2 hr 45 min and thawed in water at 40 F for 40 min. Typical cooling rates are given in the lower portion of Fig. 1. The automatic apparatus produced approximately 7 cycles of freezing and thawing per day or 50 cycles per week. week. Routine weight, length, and fundamental frequency measurements were made on all specimens. Exposure was terminated when specimens had lost 50 per cent of their initial dynamic modulus.

DISCUSSION OF DATA

The principal data are presented in Tables 3 to 7 and in Figs. 4 to 10. The tables furnish the values for the five measurements referred to earlier: dila-

TABLE 3.—DILATION IN SLOW CYCLE.

Immersion Week	Dilation, μin. per in.												Average
	Round A						Round B						
	1	2	3	4	5	6	1	2	3	4	5	6	
WET LIMESTONE													
0....	31	25	44	34	28	31	25	22	14	19	14	...	26
2....	85[a]	56[a]	110	58	62	68	64[a]	65	55	66	50	...	67
4....	...	...	132	84	62	80	...	100	80	109	88	...	92
6....	...	...	205	172	103	144	...	166[a]	134[a]	185	140	...	156
8....	...	...	292	256	155[a]	204[a]	...	...	...	288	223	...	236
10....	...	...	391	397	...	...	...	...	...	366	285	...	360
12....	...	...	474	493	...	...	...	...	...	433[a]	356[a]	...	439
14....	...	...	575	574	...	...	...	...	...	...	...	...	575
16....	...	...	626	646	...	...	...	...	...	...	...	...	636
18....	...	...	673[b]	683[b]	...	...	...	...	...	...	...	...	678
DRY LIMESTONE													
0....	0	0	0	0	0	0	0	0	0	0	0	0	0
2....	0	0	0	0	0	0	0	0	0	0	0	0	0
4....	0	0	0	0	0	0	0	0	0	0	0	0	0
6....	0	0	0	0	0	0	0	0	0	0	0	0	0
8....	0	0	0	0	0	0	11[a]	13[a]	0	0	36	0	5
10....	0	0	38	12	22	0	...	...	18[a]	28[a]	61	16	16
12....	21	12	49	6	42	23	...	...	...	...	69	19	30
14....	33[a]	26[a]	84	17	83	27	...	...	...	...	81	47	50
16....	...	...	100[a]	40[a]	86	45	...	...	...	...	91[a]	50[a]	69
18....	...	...	...	...	84[a]	34[a]	...	...	...	...	...	...	59
WET CHERT													
0....	946[a]	638[a]	613	569	696	812	794[a]	888[a]	775	820	606	606	730
2....	...	...	1120[b]	1090[b]	1350[b]	1320[b]	...	...	1531[b]	1330[b]	1150[b]	1158[b]	1256
DRY CHERT													
0....	0	0	0	0	0	0	0	0	0	0	0	0	0
2....	0	0	0	0	0	0	0	0	0	0	0	0	0
4....	0	49	0	0	0	0	0	0	0	0	0	0	4
6....	0	109	33	0	0	0	0	29	31	0	0	47	21
8....	95[a]	184[a]	153	63	81	100	80	68	100	56	49[a]	64[a]	91
10....	...	...	256	150	131	153	147	116	161[a]	103[a]	...	...	152
12....	...	...	347	240	225	254	193	170	...	...	...	...	238
14....	...	...	386[a]	272[a]	253	258	282[a]	240[a]	...	...	...	...	282
16....	...	...	...	...	252	258	...	...	...	...	...	...	255
18....	...	...	...	...	312	306	...	...	...	...	...	...	309
20....	...	...	...	...	350	338	...	...	...	...	...	...	344
22....	...	...	...	...	392[a]	385[a]	...	...	...	...	...	...	389

[a] Prism transferred to rapid cycle (ASTM Method C 291).

[b] Dynamic modulus was less than 50 per cent. Exposure terminated.

tion in Table 3, residual length change in Table 4, cumulative length change in Table 5, moisture content in Table 6, and reductions in dynamic modulus in Table 7.

The dilations were determined from typical cooling curves such as those shown in Fig. 4. Data are given for one dolomitic-limestone-concrete prism dry cured and for one wet cured. For the

TABLE 4.—RESIDUAL LENGTH CHANGE IN SLOW CYCLE.

Immersion Week	Residual Length Change, μin. per in.												Average
	Round A						Round B						
	1	2	3	4	5	6	1	2	3	4	5	6	
	WET LIMESTONE												
0	82	28	46	25	33	64	36	39	45	51	53	...	46
2	60[a]	17[a]	27	16	13	2	34[a]	43	32	51	43	...	34
4	...	...	60	33	36	72	...	72	45	52	49	...	52
6	...	...	78	52	40	90	...	102[a]	71[a]	82	80	...	74
8	...	...	129	104	78[a]	121[a]	...	...	...	98	116	...	108
10	...	...	136	119	...	...	...	...	...	125	155	...	134
12	...	...	182	152	...	...	...	...	...	146[a]	169[a]	...	162
14	...	...	195	189	...	...	...	...	...	...	...	...	192
16	...	...	210	192	...	...	...	...	...	...	...	...	201
18	...	...	243[a]	218[a]	...	...	...	...	...	...	...	...	231
	DRY LIMESTONE												
0[b]	115	97	98	108	101	84	104	116	118	120	112	104	106
2	−9	−7	16	−4	1	5	6	15	25	12	7	8	6
4	12	25	9	21	0	9	14	17	20	14	13	15	14
6	5	1	17	12	9	10	12	15	18	26	14	18	13
8	9	8	7	6	6	9	10[a]	14[a]	12	13	5	7	9
10	10	4	13	7	4	8	...	...	12[a]	10[a]	7	12	9
12	12	0	5	2	5	5	...	...	...	...	24	14	10
14	21[a]	5[a]	12	4	6	5	...	...	...	...	12	13	10
16	...	...	33[a]	27[a]	32	26	...	...	...	...	28[a]	32[a]	30
18	...	...	...	...	84[a]	34[a]	...	...	...	...	...	...	59
	WET CHERT												
0	622[a]	342[a]	384	347	464	665	458[a]	568[a]	443	596	234	461	419
2	...	...	610[c]	598[c]	794[c]	790[c]	...	...	743[c]	880[c]	532[c]	765[c]	714
	DRY CHERT												
0[b]	94	93	92	88	110	77	95	92	93	99	101	102	95
2	7	−10	−4	25	−4	12	9	6	2	3	8	6	5
4	9	2	17	23	34	35	16	13	12	17	15	27	20
6	17	49	30	19	48	24	30	37	48	23	24	24	31
8	72[a]	86[a]	87	62	87	96	55	56	66	43	40[a]	30[a]	65
10	...	...	132	65	84	91	84	74	98[a]	74[a]	...	...	88
12	...	...	169	103	108	118	94	92	...	...	...	...	114
14	...	...	194[a]	124[a]	113	116	114[a]	94[a]	...	...	...	...	126
16	...	...	...	...	120	120	...	...	...	...	...	...	120
18	...	...	...	...	131	115	...	...	...	...	...	...	123
20	...	...	...	...	126	120	...	...	...	...	...	...	123
22	...	...	...	...	146[a]	130[a]	...	...	...	...	...	...	139

[a] Prism transferred to rapid cycle (ASTM Method C 291).

[b] Expansion due largely to absorption of water.

[c] Dynamic modulus was less than 50 per cent. Exposure terminated.

first cycle of the dry-cured prism the change in length was essentially linear thoughout the range of 40 F to 0 F. No dilation was indicated.

For the second cycle there is still no evidence of dilation, but the slope of the cooling curve is much steeper, due to the water absorbed during the intervening

TABLE 5.—TOTAL CUMULATIVE CHANGE IN SLOW CYCLE.

Immersion Week	Cumulative Length Change, μin. per in.												Average
	Round A						Round B						
	1	2	3	4	5	6	1	2	3	4	5	6	
WET LIMESTONE													
0.....	82	28	46	25	32	64	36	39	45	51	52	...	46
2.....	162[a]	55[a]	93	56	57	84	80[a]	82	71	97	87	...	77
4.....	...	...	170	104	106	174	...	154	124	161	138	...	141
6.....	...	...	254	165	154	269	...	254[a]	202[a]	245	221	...	221
8.....	...	...	380	272	233[a]	392[a]	...	...	...	343	330	...	325
10.....	...	...	521	396	...	...	...	...	...	464	481	...	466
12.....	...	...	719	557	...	...	...	...	...	609[a]	641[a]	...	632
14.....	...	...	911	761	...	...	...	...	...	...	...	...	836
16.....	...	...	1128	945	...	...	...	...	...	...	...	...	1037
18.....	...	...	1370[b]	1159[b]	...	...	...	...	...	...	...	...	1265
DRY LIMESTONE													
0.....	115	95	98	108	101	84	162	160	158	156	164	172	131
2.....	218	209	224	221	222	218	248	247	267	246	253	266	237
4.....	253	246	253	261	261	250	279	286	319	276	283	314	273
6.....	258	245	270	267	264	258	293	299	337	302	299	334	286
8.....	272	253	277	274	270	267	308[a]	313[a]	353	315	308	345	296
10.....	282	257	290	281	274	275	...	...	375[a]	333[a]	323	363	305
12.....	322	279	338	313	314	298	...	...	...	...	351	384	325
14.....	349[a]	294[a]	367	330	330	317	...	...	...	...	370	403	345
16.....	...	...	404[a]	366[a]	366	347	...	...	...	...	388[a]	425[a]	383
18.....	...	...	...	...	386[a]	364[a]	...	...	...	...	...	...	375
WET CHERT													
0.....	622[a]	342[a]	384	347	464	665	458[a]	568[a]	443	597	234	461	419
2.....	...	...	977[b]	926[b]	1239[b]	1426[b]	...	...	1175[b]	1454[b]	748[b]	1218[b]	1145
DRY CHERT													
0.....	94	93	92	88	110	77	95	92	93	101	101	102	95
2.....	132	119	121	141	80	133	151	136	137	134	143	140	131
4.....	144	151	138	135	103	155	171	155	157	158	161	171	150
6.....	179	200	177	159	146	169	183	184	201	169	175	187	177
8.....	242[a]	276[a]	256	213	227	256	242	240	273	212	215[a]	221[a]	239
10.....	...	...	395	302	308	344	328	323	381[a]	291[a]	...	...	334
12.....	...	...	574	408	424	464	428	425	...	...	...	...	454
14.....	...	...	759[a]	527[a]	537	572	554[a]	519[a]	...	...	...	...	578
16.....	...	...	...	...	662	684	...	...	...	...	...	...	673
18.....	...	...	...	...	791	805	...	...	...	...	...	...	798
20.....	...	...	...	...	923	929	...	...	...	...	...	...	926
22.....	...	...	...	...	1060[a]	1053[a]	...	...	...	...	...	...	1057

[a] Prism transferred to rapid cycle (ASTM Method C 291).

[b] Dynamic modulus was less than 50 per cent. Exposure terminated.

two weeks of immersion. Note that the slope for the fourth cycle is the same as that for the second. The first clear evidence of dilation occurred during the sixth cycle (38 μin. per in.) and increased to 84 μin. per in. dilation at the eighth cycle. (See Table 3 for specimen No. 3, round A, dry-cured limestone concrete.)

TABLE 6.—MOISTURE CONTENT OF CONCRETES IN SLOW CYCLE.

Determined by drying specimens to constant weight at 230 F at end of exposure.

Immersion Week	Moisture Content, Per Cent of Oven Dry Weight												Average
	Round A						Round B						
	1	2	3	4	5	6	1	2	3	4	5	6	
						WET LIMESTONE							
0	8.33	8.29	8.28	8.33	8.36	8.15	8.64	8.67	8.85	8.63	8.61	...	8.47
2	8.56[a]	8.47[a]	8.49	8.53	8.56	8.34	8.86[a]	8.86	9.04	8.44	8.83	...	8.67
4	...	...	8.58	8.62	8.64	8.45	...	8.99	9.16	8.94	8.93	...	8.70
6	...	...	8.66	8.69	8.71	8.51	...	9.05[a]	9.22[a]	9.01	9.01	...	8.86
8	...	...	8.75	8.74	8.75[a]	8.56[a]	...	...	...	9.07	9.06	...	8.82
10	...	...	8.82	8.81	...	...	...	...	...	9.13	9.12	...	8.97
12	...	...	8.88	8.90	...	...	...	...	...	9.16[a]	9.17[a]	...	9.03
14	...	...	8.93	8.97	...	...	...	...	...	...	...	...	8.95
16	...	...	8.98	9.02	...	...	...	...	...	...	...	...	9.00
18	...	...	9.06[b]	9.05[b]	...	...	...	...	...	...	...	...	9.06
						DRY LIMESTONE							
0	5.09	5.17	5.11	5.07	5.01	5.12	4.99	4.99	5.02	5.00	4.92	4.88	5.03
2	7.57	7.52	7.45	7.45	7.41	7.55	7.65	7.64	7.71	7.64	7.54	7.61	7.56
4	7.70	7.64	7.61	7.63	7.54	7.68	7.86	7.83	7.90	7.82	7.71	7.78	7.73
6	7.82	7.77	7.77	7.73	7.64	7.80	7.92	7.93	8.00	7.92	7.81	7.88	7.84
8	7.87	7.83	7.86	7.78	7.71	7.84	8.02[a]	7.99[a]	8.06	7.98	7.87	7.94	7.90
10	7.92	7.87	7.91	7.78	7.75	7.90	...	...	8.10[a]	8.01[a]	7.90	8.07	7.92
12	7.95	7.92	7.94	7.81	7.78	7.93	...	...	...	...	7.92	8.07	7.92
14	7.97[a]	7.93[a]	7.96	7.84	7.87	7.97	...	...	...	...	7.93	8.08	7.94
16	...	...	8.02[a]	7.87[a]	7.89	7.97	...	...	...	...	7.97[a]	8.11[a]	7.97
18	...	...	...	...	7.94[a]	8.00[a]	...	...	...	...	...	...	7.97

WET CHERT													
0	10.55[a]	10.33[a]	9.75	9.67	9.83	9.91	10.30[a]	10.17[a]	10.15	10.07	10.04	9.78	10.05
2[b]	...	...	10.07	9.98	10.16	10.25	...	...	10.47	10.36	10.30	10.03	10.20
DRY CHERT													
0	6.27	6.39	6.27	6.32	6.27	5.96	6.22	6.09	6.07	6.13	6.05	6.27	6.19
2	8.15	8.31	8.13	8.27	8.24	7.94	8.34	8.18	8.20	8.33	8.36	8.44	8.24
4	8.31	8.50	8.33	8.42	8.44	8.05	8.59	8.41	8.45	8.55	8.58	8.69	8.44
6	8.47	8.65	8.48	8.54	8.61	8.18	8.71	8.53	8.57	8.64	8.70	8.81[a]	8.57
8	8.63[a]	8.77[a]	8.68	8.70	8.79	8.34	8.85	8.67	8.74	8.79	8.81[a]	8.94	8.73
10	...	...	8.85	8.87	8.96	8.46	9.07	8.82	8.88[a]	8.93[a]	...	...	8.86
12	...	...	8.96	8.98	9.04	8.50	9.10	8.94	...	...	...	...	8.92
14	...	...	9.02[a]	9.01[a]	9.08	8.55	9.17[a]	9.02[a]	...	...	...	...	8.98
16	...	...	...	...	9.16	8.61	...	...	...	...	...	...	8.89
18	...	...	...	...	9.19	8.64	...	...	...	...	...	...	8.92
20	...	...	...	...	9.22	8.67	...	...	...	...	...	...	8.95
22	...	...	...	...	9.27[a]	8.70[a]	...	...	...	...	...	...	8.99

[a] Prism transferred to rapid cycle (ASTM Method C 291).

[b] Dynamic modulus was less than 50 per cent. Exposure terminated.

TABLE 7.—REDUCTION IN RELATIVE *E* IN SLOW CYCLE.

Immersion Week	Reductions in Relative *E*, per cent												Average
	Round A						Round B						
	1	2	3	4	5	6	1	2	3	4	5	6	
	WET LIMESTONE												
0....	9	8	9	6	7	9	1	5	4	6	5	...	6
2....	8[a]	8[a]	9	4	6	9	1[a]	6	3	6	5	...	6
4....	...	...	13	8	7	10	...	9	4	7	6	...	8
6....	...	...	10	4	2	7	...	10[a]	5[a]	7	6	...	6
8....	...	...	17	8	6[a]	10[a]	...	...	...	9	11	...	10
10....	...	...	19	12	...	...	...	...	...	13	13	...	14
12....	...	...	28	18	...	...	...	...	...	17[a]	15[a]	...	19
14....	...	...	35	23	...	...	...	...	...	...	...	...	29
16....	...	...	38	29	...	...	...	...	...	...	...	...	33
18....	...	...	62[b]	58[b]	...	...	...	...	...	...	...	...	60
	DRY LIMESTONE												
0....	8	6	9	5	7	9	6	8	8	8	5	2	7
2....	8	7	7	6	8	9	6	8	9	8	4	8	7
4....	5	6	5	3	5	7	5	4	7	6	3	7	5
6....	2	2	1	1	1	4	4	3	8	4	3	6	3
8....	1	1	1	0	0	9	3[a]	3[a]	7	3	2	5	2
10....	1	2	2	0	2	4	...	...	3[a]	1[a]	1	2	2
12....	2	2	2	1	0	4	...	...	...	...	0	2	2
14....	2[a]	1[a]	1	1	0	1	...	...	...	...	1	2	1
16....	...	...	3[a]	1[a]	1	3	...	...	...	...	0[a]	2[a]	2
18....	...	...	...	...	0[a]	1[a]	...	...	...	...	...	...	1
	WET CHERT												
0....	47[a]	34[a]	28	28	29	46	29[a]	31[a]	52	43	20	28	35
2....	...	...	59[b]	61[b]	64[b]	69[b]	...	...	65[b]	67[b]	72[b]	66[b]	65
	DRY CHERT												
0....	2	5	1	4	5	3	1	0	0	1	1	1	2
2....	0	0	0	0	1	0	0	0	0	0	0	0	0
4....	0	0	0	0	0	0	0	0	0	0	0	0	0
6....	0	1	0	0	1	0	0	0	2	0	0	0	0
8....	6[a]	6[a]	4	1	8	0	2	0	8	0	0[a]	0[a]	3
10....	...	...	18	9	10	3	8	5	13[a]	3[a]	...	...	9
12....	...	...	28	17	19	17	11	10	...	...	...	...	17
14....	...	...	42[a]	28[a]	28	27	17[a]	16[a]	...	...	...	...	26
16....	...	...	...	...	26	25	...	...	...	...	...	...	26
18....	...	...	...	...	28	28	...	...	...	...	...	...	28
20....	...	...	...	...	32	32	...	...	...	...	...	...	32
22....	...	...	...	...	36[a]	35[a]	...	...	...	...	...	...	36

[a] Prism transferred to rapid cycle (ASTM Method C 291).

[b] Dynamic modulus was less than 50 per cent. Exposure terminated.

The wet-cured concrete showed dilation on the first cycle. Dilation increased progressively with further immersion. On the eighth cycle, after 14 weeks of immersion, a dilation of 575 μin. per in was measured and the dynamic modulus has been reduced 10 per cent.

Typical cooling curves for the chert

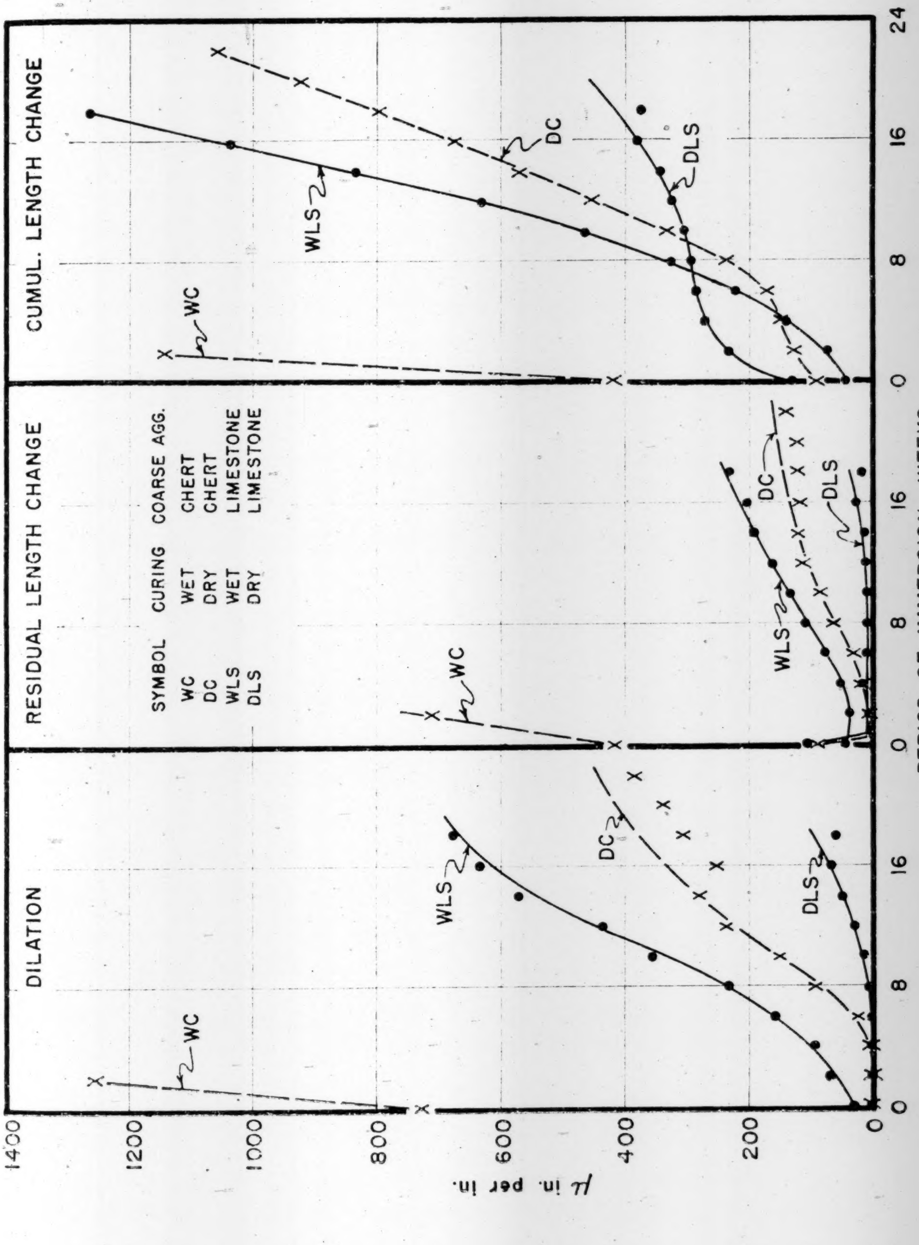

Fig. 5.—Effect of Immersion on Length Changes in the Slow Cycle.

concrete (not shown) were similar except that given dilations occurred after shorter periods of immersion. Wet-cured chert concrete showed dilations of 550 to 950 μin. per in. on the first cycle and reductions in dynamic modulus of the order of 20 per cent.

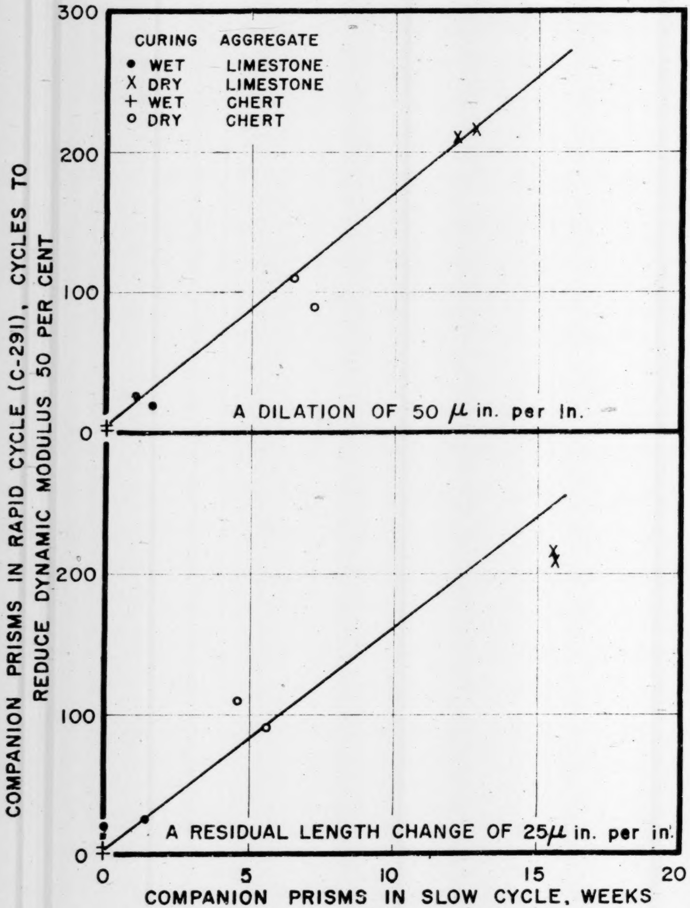

FIG. 6.—Comparison of Slow and Rapid Cycles.

Period of Immersion Versus Dilation and Length Change:

Figure 5 shows the effect of period of immersion on dilation, residual length change, and cumulative length change. Note that the wet-cured concretes showed dilation on the first freezing while 4 to 8 weeks elapsed before dilation occurred for the dry-cured concrete. The curves for the residual length changes are similar in shape to those for dilation, except that the dry-cured concrete showed a residual length change on the first cycle, due to the expansion resulting from the water absorbed during thawing.

Cumulative length change and, to a lesser extent, residual length change are due to the combined separate effects of freezing damage, absorption of water, and the gradual growth due to cycles of heating and cooling. For this reason, cumulative length change is a less useful measure of the effects of freezing than dilation or residual length change (see Fig. 5).

As pointed out earlier, the period of immersion required to produce a significant dilation may be a good index of durability. Interpolation of average data from Table 4 shows a dilation of 50 μin. per in. to have been attained: (1) in 1 week for wet-cured limestone concrete and 12 weeks for the dry cured, and (2) in 0 weeks (first freezing) for wet-cured chert concrete and 7 weeks for dry cured. Similar comparisons may be made for dilations as small as about 25 μin. per in., but at this point the variations inherent in the test are as large as the values measured.

Slow Versus Rapid Cycle:

A comparison of the durability of concrete as measured on two groups of specimens from the same batch, one tested in the slow cycle and the other in the rapid cycle, is given in Fig. 6. For the slow cycle, two measures are shown: (1) the weeks of immersion required to produce a residual length change of 25 μin. per in. and (2) the period to produce a dilation of 50 μin. per in. In the rapid cycle, the criterion is the number of cycles of freezing and thawing required to reduce the dynamic modulus 50 per cent. For the few data available, an excellent relationship is shown—the number of fast cycles are directly proportional to the number of weeks of immersion in the slow cycle.

When large residual length changes were measured on the first cycle but not on the second, they were judged to have been due to the absorption of water rather than to freezing damage and, therefore, neglected in preparing Fig. 6.

Dilation Versus Residual Length Change:

Figure 6 suggests that measurements of dilation and residual length change in the slow cycle may be equally significant—or, at the least, show substantially the same agreement with the rapid test. If that is so and if the measurement of dilation can be omitted, the test procedure is greatly simplified; the laborious measurements to establish the cooling curve would not be required.

Further information on this question is given in Fig. 7. While there is considerable scatter of the points, the relationship is reasonably good when the high values of dilation and length change are taken into account. The residual length change was about one third to one half of the corresponding dilation.

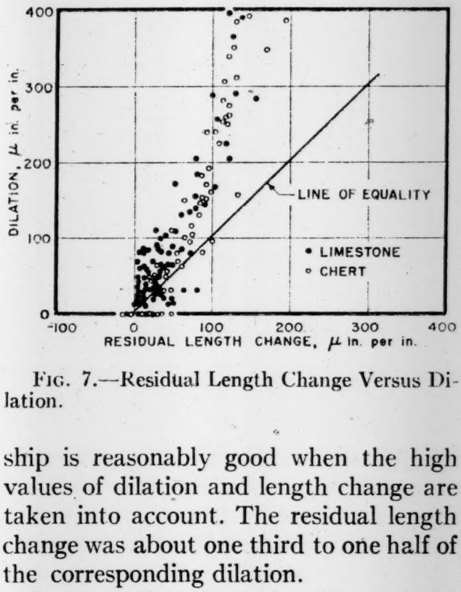

Fig. 7.—Residual Length Change Versus Dilation.

It seems clear from the data and the earlier discussion that significant information on the resistance of the specimen is secured at 50 μin. per in. or less. In this region the relationship is highly variable. Dilations of less than 20 μin. per in. were difficult to detect. Dilations were measured over the full length of the prism using the vertical comparator; and residual length changes were measured with a Whittemore strain gage over a central 10-in. gage length. Random

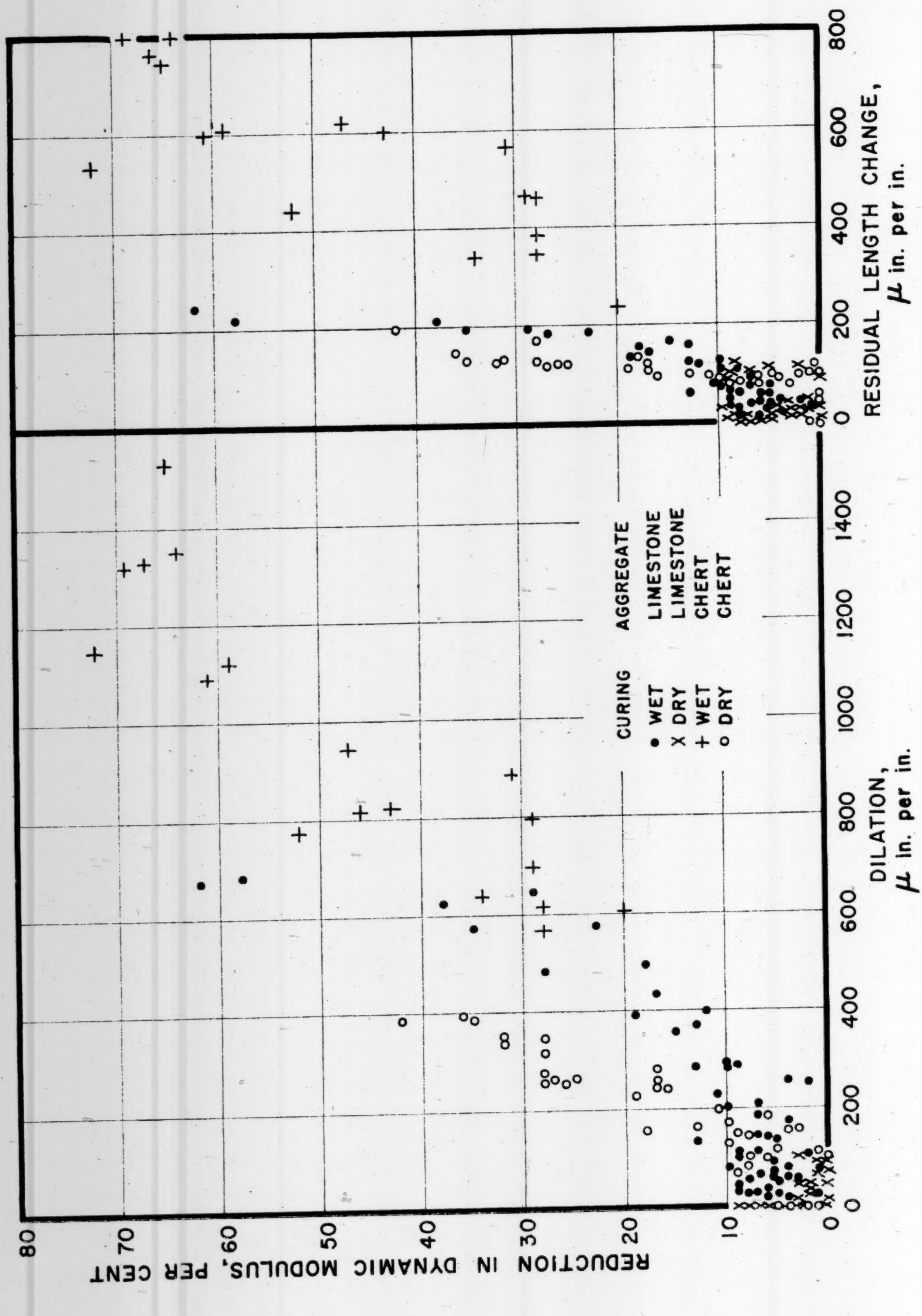

FIG. 8.—Relation of Dilation and Residual Length Change to Reduction in Relative Modulus of Elasticity in Slow Cycle.

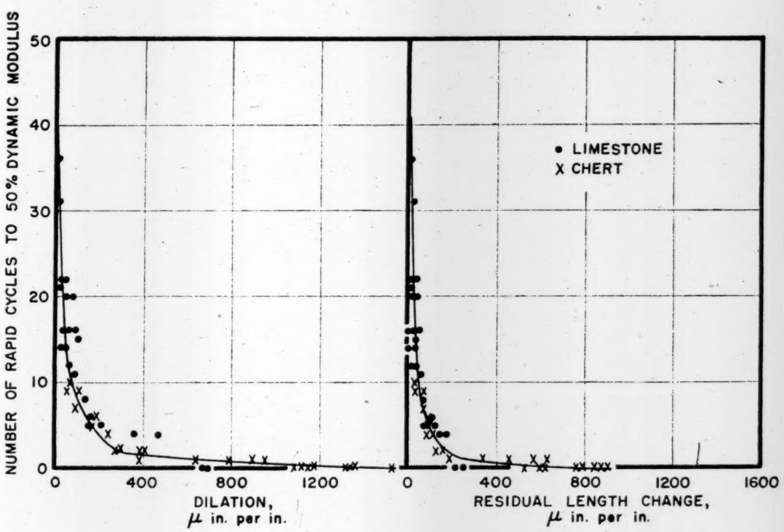

FIG. 9.—Resistance to Rapid Cycle After Exposure to Slow Cycle.

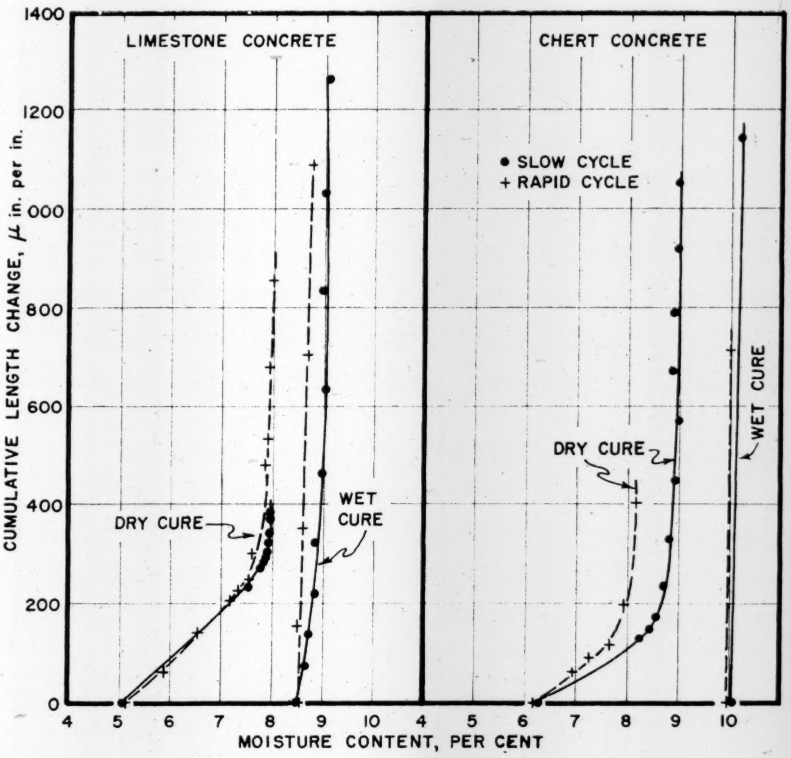

FIG. 10.—Relationships Between Moisture Content and Cumulative Length Change.

location of a few bad pieces of aggregate which are the first to become critically saturated and the fact that the ends of the prism were more severely affected than the central portions increased the differences due to method of measurement. More data are required before conclusions can be drawn on this point.

Figure 8 compares measurements of dynamic modulus with those of dilation and residual length change. The data indicate a fairly good relationship; dilation and residual length changes are accompanied by reductions in dynamic modulus. They also show, however, that length changes which are considered to be significant have occurred before the dynamic modulus of elasticity had been reduced as much as 10 per cent.

Rapid Cycle Tests on Slow Cycle Specimens:

As a measure of the amount of damage resulting from the slow cycle, specimens which had attained various dilations were transferred to the standard rapid cycle. Figure 9 shows both dilation and residual length change as abscissa. The ordinate gives the number of rapid cycles required to produce failure (loss of 50 per cent dynamic modulus) of specimens which had already been subjected to the slow cycle and had attained the length changes indicated. Note that resistance to the rapid cycle was sharply reduced when specimens had dilated well under 50 μin. per in. in the slow cycle or, equivalently, attained residual length changes under 25 μin. per in.

Moisture Content Versus Total Length Change:

Although cumulative length change is not considered a good indicator of specimen condition, it provides a convenient method of comparing the effects of moisture content on specimens in the slow and rapid cycles (see Fig. 10). The curves show that the wet-cured specimens were at, or very near, critical moisture-content levels at the start of exposure, and large total length changes developed rapidly with the absorption of very little additional water. The dry-cured concretes first expanded on account of the absorption of water, but at some point critical moisture contents were developed and, again, expansion increased rapidly with little change in moisture content.

The curves for companion specimens in the slow and rapid cycles are remarkably similar. The dry-cured limestone concretes developed critical saturation at the same moisture content in both exposures. The dry-cured chert concrete failed at a lower moisture content in the rapid cycle than companion specimens in the slow cycle, probably because of the early saturation of the more absorptive particles of the chert in chert gravel and prompt exposure to a large number of cycles of freezing and thawing.

CONCLUSIONS

These data, based on tests of air-entrained concretes made with two dissimilar aggregates, both having poor resistance to freezing and thawing in concrete, and cured by two methods, appear to support the following conclusions:

1. Dilation and residual length change are indications of a moisture condition at which concretes are susceptible to freezing damage. The period of immersion required to produce measurable dilation is a significant measure of the resistance of concrete to freezing and thawing.

2. In this investigation measurements of dilations as low as 25 to 30 μin. per in. presented difficulties due to a number of factors including instrumentation and problems of temperature control. More refinement in controls is indicated.

3. The measurement of residual length change alone may afford an adequate measure of specimen condition.

4. The data demonstrate the beneficial effects of a short period of drying before exposure to freezing and thawing. The moisture content of specimens given a relatively short period of air drying was restored to critical levels only after an extended period of soaking.

5. For these tests there was good correlation between the results of the slow cycle as evaluated by dilation and those for companion specimens in the rapid cycle as evaluated by loss in dynamic modulus. Similar comparisons should be made with the three other ASTM standard procedures, using aggregates which would produce a greater range in resistance to freezing and thawing.

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HIGH-TEMPERATURE BEHAVIOR OF ALUMINOUS CEMENT CONCRETES CONTAINING DIFFERENT AGGREGATES*

By N. G. Zoldners,[1] V. M. Malhotra,[1] and H. S. Wilson[1]

Synopsis

Changes in physical properties of over 350 concrete test specimens, made with aluminous cement and expanded shale, anorthosite, ilmenite, and phonolite aggregates, were studied after exposure to temperatures ranging from 100 to 1100 C. It was found that the aluminous cement concrete loses its strength rapidly in the range of temperatures investigated. Expanded shale aggregate concrete showed the greatest stability, retaining about 20 per cent of its original compressive strength after firing at 1000 C, whereas concrete made with phonolite aggregate was the least heat-resistant of the four types investigated.

The fired concrete specimens were tested for strength and examined petrographically. Probable causes of reduction in strength due to exposure to elevated temperatures are discussed briefly.

Concrete for industrial installations exposed to elevated temperatures requires heat-resistant materials. The need for such materials is particularly great in the chemical and metallurgical industries and for the thermal shieldings of nuclear power plants.

Concretes made with hydraulic cements and conventional aggregates are designed for service in normal environmental conditions. The heat-resistance of these concretes is greatly limited and is governed mainly by the types of cement and aggregate used.

A literature survey revealed that, until recently, little information was available on the effect of dry heat at relatively low temperatures (100 to 300 C) on the properties of concrete made with aluminous cement.

One of the earliest publications in this field was a study by Miller and Faulkner (**1**),[2] indicating a surprising reduction in strength of aluminous cement concrete after heating.

Recently, relevant studies were published by the Universal Atlas Cement Division of United States Steel Corp., Research Laboratories (**2**,**3**), and by the Lafarge Aluminous Cement Co. Ltd., London, England (**4**–**6**).

Considerable work has been carried out in Russia during the past five years by the Institute of Scientific Research for Concrete and Reinforced Concrete, at the Academy of Building and Architecture (**7**–**10**).

Scope of Research

The purpose of this study was to investigate the physical properties of aluminous cement concretes after exposure for 1 hr to temperatures ranging from 100 to 1000 C. Four aggregates were used having widely different mineralogical and physical properties. These consisted of a manufactured lightweight aggregate (expanded shale), a heat-resistant coarse-grained basic igneous rock (anorthosite), a commercially produced heavy aggregate (ilmenite), and a fine-grained feldspathoidal igneous rock (phonolite) exhibiting some unusual properties.

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* Presented at the Sixty-sixth Annual Meeting of the Society.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

TABLE 1.—CHEMICAL ANALYSES OF ALUMINOUS AND PORTLAND CEMENTS.

Chemical Constituents (Oxides)	Aluminum Cement Used,[a] per cent	Typical Aluminous Cements from Different Countries,[b] per cent			Type I Portland Cement,[c] per cent
		England	Germany	U. S. A.	
SiO_2	4.50	4 to 5	5 to 8	8 to 9	19 to 23
Al_2O_3	39.74	38 to 40	48 to 51	40 to 41	5 to 8
Fe_2O_3	9.98	8 to 10	0.1	5 to 6	2 to 4
CaO	32.52	36 to 39	39 to 42	36 to 37	62 to 66
MgO	0.12	1.0	1.0	1.0	1 to 4
So_3	0.13	0.1	0.5	0.2	1 to 2.5
FeO	5.40	5 to 7	1.0	5 to 6	
TiO_2	2.03	2.0	1.5	2.0	

[a] Mines Branch Internal Report MS-AC 62-293.

[b] Reference (**6**), p. 41.

[c] R. F. Blanks and H. L. Kennedy, *The Technology of Cement and Concrete*, Vol. 1, 1955, p. 28.

TABLE 2.—GENERAL PETROGRAPHIC FEATURES OF AGGREGATES.
After J. A. Soles.

Aggregate	Petrography
Expanded shale	Medium gray, externally rusty, low density, bloated fragments showing primary shale banding; highly vesicular, fused appearance.
Anorthosite	Medium- to coarse-grained (1 to 10 mm), blue-gray rock, consisting of plagioclase $Ab_{50}An_{50}$ (95 per cent) and scattered crystals of uralitized olivine (5 per cent). Relict crystals of olivine are surrounded by an inner lining of enstatite and an outer coat of chloritized hornblende.
Ilmenite rock	Medium- to coarse-grained (2 to 10 mm), consisting of crystals of black ilmenite (85 per cent), granules of plagioclase $Ab_{50}An_{50}$ (10 per cent), and spinel (1 per cent), books and layers of biotite (1 per cent), and green mica or chlorite (2 per cent), and grains of pyrite (1 per cent). The green mica surrounds and fills cracks in the plagioclase.
Phonolite (tinguaite)	Fine-grained (1 to 3 mm), light-gray rock, occasional dark bands. The approximate composition is: K feldspar (30 per cent), nepheline (25 per cent), natrolite (20 per cent), albite (15 per cent), and acmite (10 per cent). The rock has a microscopic trachytic texture.

This study is part of an extensive research project (**11–13**) being undertaken by the Mines Branch, Department of Mines and Technical Surveys, Ottawa, Canada, to determine the effect of different types of cements and aggregates on concrete exposed to dry heat at elevated temperatures. As part of this program, the heat-resistant properties of some of the rocks being used in this study as aggregate were also investigated (**14,15**). Other thermal properties, such as fusion temperature and thermal elongation, have been studied on a wide variety of rocks by others at the Mines Branch in the course of their investigations.

TEST MATERIALS

Cement:

A well-known commercially available brand of aluminous, or calcium aluminate, cement was used in this investigation.

The chemical composition of this type of cement is entirely different from that of portland cement. Table 1 gives the chemical composition of the cement used and compares it with typical aluminous cements from other countries, and with normal portland cement type I.

TABLE 3.—PHYSICAL PROPERTIES OF AGGREGATES.

Properties	Expanded Shale		Anorthosite		Ilmenite		Phonolite	
	Coarse Aggregate	Fine Aggregate	Coarse Aggregate	Fine Aggregate	Coarse Aggregate	Fine Aggregate	Coarse Aggregate	Fine Aggregate
Specific gravity, bulk, saturated, surface-dry	1.17	1.83	2.77	2.71	4.65	4.67	2.54	2.55
Absorption, per cent by weight	10.60	...	0.40	0.50	0.30	0.50	0.40	0.50
Mix proportions, per cent by weight..................	40	60	57	43	60	40	52	48

The principal cementitious constituent in commercial aluminous cements is monocalcium aluminate ($CaO \cdot Al_2O_3$) **(3,16–18)**, whereas in portland cements the most important are tricalcium and dicalcium silicates.

Standard specifications for aluminous cements have been drawn up in several European countries. However, there is no uniformity in the chemical characteristics required by these specifications. There is no standard specification for this type of cement in the United States.

Cement used in this work was of a dark gray color because of the higher iron content, and was coarser ground than type I portland cement.

Aggregates:

Graded fine and coarse fractions were prepared from each of the four types of aggregates used. These are described below:

1. Expanded shale was manufactured in a lightweight aggregate plant in Ontario. The aggregate was produced in a rotary kiln from a gray Carlsbad shale contaminated slightly by irregularly spaced bands of impure limestone. Coarse material was received as produced, but fines were crushed. Grading was done by screening.

2. Anorthosite was obtained from a building stone quarry in Quebec. The material was received as crushed rock with a maximum size of about 6 in. The blue-gray rock was coarse-grained (1 cm) and consisted principally of plagioclase crystals and altered olivine. The material was crushed and screened into coarse and fine aggregate fractions.

3. Ilmenite rock was supplied by a commercial producer of heavy aggregate from a quarry in Quebec. The crushed rock was received in two sizes, less than 1 in., retained on No. 4 sieve, and passing No. 4 sieve. It consisted predominantly of ilmenite, $FeTiO_3$, which is black in color and has a metallic luster.

4. An igneous rock, traded as "trap rock," was received from a commercial

producer in Quebec. It was supplied as coarse aggregate in graded sizes; fine aggregate had to be obtained by crushing and screening. This fine-grained gray rock was identified as phonolite (tinguaite), which is composed essentially of alkali feldspar, nepheline, natrolite (alteration product of nepheline), and soda pyroxene (19).

TABLE 4.—GRADING OF AGGREGATES.

Coarse Aggregates					Fine Aggregates				
Sieve Size	Per Cent Passing				Sieve Size	Per Cent Passing			
	Expanded Shale	Anorthosite	Ilmenite	Phonolite		Expanded Shale	Anorthosite	Ilmenite	Phonolite
1-in.	...	100.0	100.0	...	No. 4	100.0	100.0	100.0	100.0
¾-in.	100.0	95.0	95.0	100.0	No. 8	85.0	92.0	90.0	90.0
½-in.	65.0	65.0	65.0	56.0	No. 16	70.0	74.4	67.5	67.5
⅜-in.	30.0	35.0	35.0	37.5	No. 30	45.0	52.2	42.5	42.5
No. 4	0.0	0.0	0.0	0.0	No. 50	30.0	31.4	20.0	20.0
					No. 100	20.0	10.8	6.0	6.0

TABLE 5.—CONCRETE MIX DATA.

Type of Concrete Aggregate	Average Mix Proportions, per cu yd					
	Cement, lb	Saturated Surface-Dry Aggregates		Water, lb	Admixture, oz	W/C[a] ratio
		Coarse, lb	Fine, lb			
Expanded shale	480	704	1041	346	Darex-6	0.718
Anorthosite	482	1828	1346	342	nil	0.710
Ilmenite	471	3323	2224	333	nil	0.706
Phonolite	479	1550	1445	347	nil	0.725

[a] W/C = water-cement ratio, by weight.

A detailed petrographic study was made on each of the four types of aggregates used (Table 2).

Some physical properties of the four aggregates and the proportions of coarse to fine fractions in the concrete mixes are shown in Table 3.

The gradings of the coarse and fine aggregate used for the various materials are shown in Table 4.

CONCRETE

In order to make the needed number of test specimens, a minimum of six concrete batches was required for each type of aggregates. A total of 24 batches was prepared between January and November, 1962. The mixing and molding of test specimens were done in the mornings between 9:00 and 11:00.

Mix Design:

The same basic mix design was used in all mixes. It was calculated by the absolute volume method in accordance with the following conditions:

1. The cement content was kept constant at 480 lb per cu yd of concrete for all test mixes.
2. The water-cement ratio (*W/C*) and

the ratio of coarse to fine aggregate were kept constant for each type of concrete.

3. Air-entraining agent (Darex) was used for the expanded shale mix. All other mixes were non-air-entrained.

TABLE 6.—PROPERTIES OF FRESH CONCRETE.

Type of Concrete Aggregate	*W/C* ratio	Slump, in.	Air, per cent	Unit Weight, lb per cu ft
Expanded shale..	0.718	1¾	5.0[a]	94.3
Anorthosite......	0.710	1½	3.1	148.0
Ilmenite.........	0.706	1	0.7	234.9
Phonolite........	0.725	1	1.2	141.5

[a] Air-entraining agent used.

Apart from the air-entraining agent, no other admixtures such as plasticizers or densifiers were used for any of the concretes, because little is known about the effect of such additives on aluminous cement concretes (20).

Preparation of Concrete:

Each mix was prepared in 2-cu-ft batches, using a counter-current concrete mixer. The total mixing time for each batch was 6 min.

The room-dry coarse and fine aggregate was soaked in water 24 hr prior to its use.

The actual mix proportions used in these test mixes are given in Table 5.

Characteristics of Fresh Concrete:

The various concretes were dark in color, due to the darker shade of the aluminous cement. The considerable harshness of the mixes was significant, because of the relatively coarse grind of the cement (16). The ilmenite and anorthosite concretes were slightly undersanded and their plasticity was rather low. The expanded shale concrete showed a tendency to lose slump rapidly, although the aggregates were soaked for 24 hr.

Some of the measured properties of the fresh concrete, namely, the slump, air content, and unit weight, were determined using ASTM standard testing methods.[3] The test results are summarized in Table 6.

TABLE 7.—SUMMARY OF TEST SPECIMENS.

Type of Concrete Aggregate	Number of Batches	Number of Specimens for Each Type of Concrete			Total Number of Specimens
		4 by 8 in. Cylinders	3½ by 4 by 16 in. Beams	3 by 13 by 17½ in. Slabs	
Expanded shale..........	5	45	45	...	90
	1	...	...	2	2
Anorthosite.............	5	45	45	...	90
	1	...	...	2	2
Ilmenite................	5	45	45	...	90
	1	...	...	2	2
Phonolite...............	5	45	45	...	90
	1	...	...	2	2
Total	24	180	180	8	368

Test Specimens:

Eighteen test specimens, consisting of nine 4 by 8-in. cylinders and nine 3½ by 4 by 16-in. beams, were cast from each mix. Three cylinders and three beams from each mix were used as reference specimens. Six cylinders and six beams were kept for two firing exposures, using three specimens of each kind for every firing. To provide test specimens for ten temperature exposures, five batches were required.

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[3] Method of Test for Slump of Portland Cement Concrete (C 143), Method of Test for Air Content of Freshly Mixed Concrete by the Pressure Method (C 231), Method of Test for Air Content of Freshly Mixed Concrete by the Volumetric Method (C 173), and Method of Test for Weight per Cubic Foot, Yield, and Air Content (Gravimetric) of Concrete (C 138), 1961 Book of ASTM Standards, Part 4.

In addition to the batches for fire test specimens, one additional batch was made for each type of aggregate to provide two 3 by 13 by $17\frac{1}{2}$-in. slabs for thermal conductivity studies.

Table 7 gives a summary of various test specimens prepared from each type of concrete.

TABLE 8.—TEMPERATURE IN THE CASTING ROOM.

Type of Concrete Aggregate	Date of Mixing	Temperature Range in Casting Room for the First 24 hr, deg C
Anorthosite	Jan. 11–30, 1962	17 to 28
Phonolite	April 25–May 4, 1962	20 to 27
Expanded shale	Sept. 11–19, 1962	20 to 24
Ilmenite	Nov. 13–21, 1962	18 to 22

TABLE 9.—CONCRETE DENSITY AND CYLINDER COMPRESSIVE STRENGTH.

Type of Concrete Aggregate	7-Day, Saturated, Surface-Dry	Cylinders in Room-Dry Condition, Before Firing				
	Density,[a] lb per cu ft	Age, days	Moisture, per cent	Density,[a] lb per cu ft	Strength,[b] psi	Coefficient of Variation, per cent
Expanded shale	95.0	120	4.2	86.0	2110	4.5
Anorthosite	150.9	180	2.6	145.3	2720	6.9
Ilmenite	236.3	95	1.7	232.3	5960	6.7
Phonolite	142.8	118	3.0	136.9	2680	11.7

[a] Mean of 5 × 9 = 45 cylinders.
[b] Mean of 5 × 3 = 15 cylinders.

Molding and Curing of Test Specimens:

The test cylinders were cast in aluminum and the beams in heavy brass molds with $\frac{3}{8}$-in. side plates and $\frac{1}{2}$-in. end plates. Stainless steel reference plugs were cast in each end of the beam specimens for length measurements.

Test specimens were molded by placing concrete in the molds in two layers and vibrating them on a vibrating table for 10 and 20 sec for each layer. These vibrating times had to be doubled for the expanded shale mixes because of the poor workability of that concrete. When all test specimens were cast, each mold was topped with a glass plate and covered by water-saturated burlap. The molded specimens were left in the casting room for 24 hr, during which period the ambient room temperature varied from 17 C to 28 C. The temperature was recorded three times during the first six hours, and once the next morning. The temperature ranges recorded for all mixes are shown in Table 8.

At the end of the initial curing period, the test specimens were removed from the molds and transferred immediately to the moist curing room at a temperature of 23 ± 2 C and 95 per cent relative humidity.

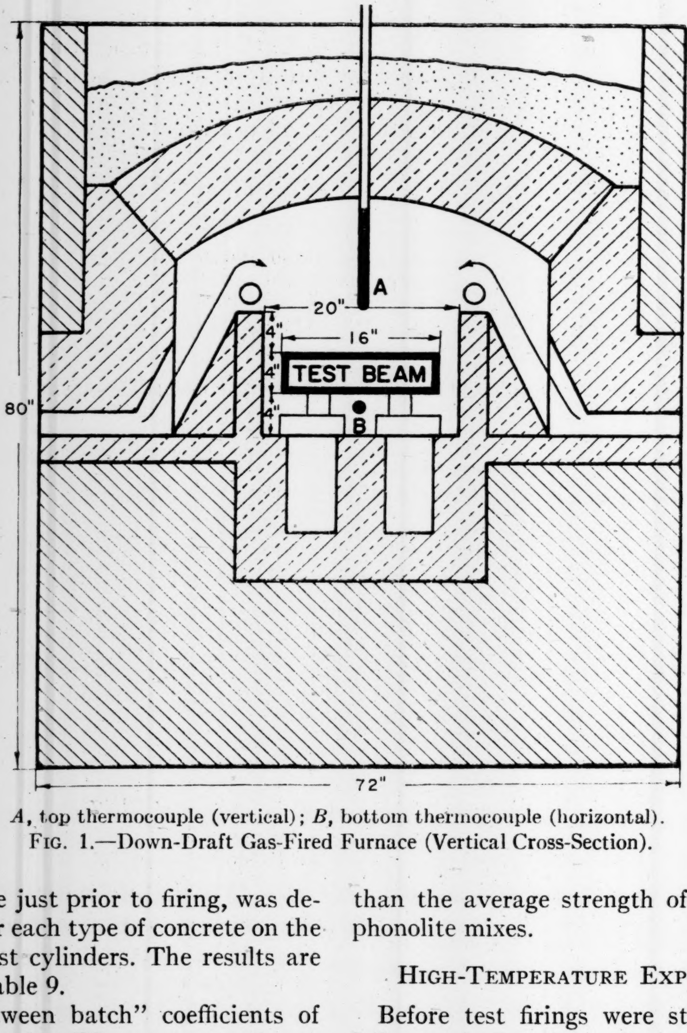
After 7 days, the specimens were removed to a storage room, the ambient temperature of which was 24 ± 2 C and the relative humidity 65 ± 5 per cent.

Test specimens remained in the dry storage until being fire tested.

PROPERTIES OF HARDENED CONCRETE

The density of the concrete in the saturated, surface-dry condition after 7 days of initial moist curing, and of the room-dry concrete just prior to firing, was determined for each type of concrete on the reference test cylinders. The results are shown in Table 9.

The "between batch" coefficients of variation (21) for different types of concrete, as calculated for the compressive strengths of the cylinders before firing, are also given in Table 9. These are 4.5, 6.9, and 6.7 per cent for expanded shale, anorthosite, and ilmenite concrete mixes, respectively, reflecting excellent to good (22) uniformity among the mixes. The high value of 11.7 per cent for the coefficient for phonolite concrete mixes was due to the low compressive strength of one of the mixes, which was 730 psi lower than the average strength of the other phonolite mixes.



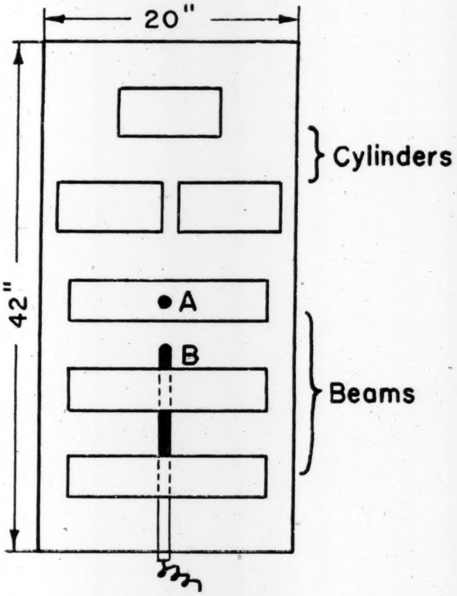
A, top thermocouple (vertical); *B*, bottom thermocouple (horizontal).
FIG. 1.—Down-Draft Gas-Fired Furnace (Vertical Cross-Section).

HIGH-TEMPERATURE EXPOSURE

Before test firings were started, the 7-day moist-cured specimens were air-dried at the ambient room temperature. The drying period varied from 95 days for ilmenite to 180 days for anorthosite concrete.

During this period, the moisture content of the concretes from the saturated, surface-dry condition was appreciably reduced and ranged from 1.7 per cent for ilmenite concrete to 4.2 per cent for expanded shale concrete.

The preconditioning of fire-test specimens by air-drying was necessary to avoid spalling damage of concrete, which may be caused by pressure of the steam driven out during firing tests. Test specimens designated for fire tests were marked with a fire-resistant green chrome paint. All firings were conducted in the furnace described below.



A, top thermocouple (vertical); *B*, bottom thermocouple (horizontal).

FIG. 2.—Arrangement of Test Specimens in Furnace—Plan View.

Description of Furnace:

A gas-fired, down-draft furnace, with internal dimensions of 20 in. wide, 42 in. deep and 24 in. high, was used to fire the concrete test specimens. It was heated by six burners, three on each side. Heating was controlled by changing the size of the burner tips, by regulating the flow of gas and of natural draft to each burner, and by varying an induced draft, using a damper in the exhaust flue. This created a reducing atmosphere in the furnace.

Two thermocouples connected to a recording pyrometer were used to record temperature. One was situated vertically in the center of the heating chamber of the furnace, above the test specimens, and the other horizontally, immediately below the specimens. A vertical section of the furnace is shown in Fig. 1.

Arrangement of Specimens in Furnace:

Three cylinders and three beam test specimens were fired at one time. The cylinders were laid on their sides at the back of the heating chamber and the beams were set on their $3\frac{1}{2}$ by 16-in. faces in front of the cylinders as shown in Fig. 2.

Preliminary Firing:

A preliminary firing with dummy test specimens was made to determine the heating rate of the furnace up to 1000 C. It was found to be close to the standard heating rate specified by ASTM methods E 119.[4] The recorded heating curves obtained in the preliminary firing, and the standard time-temperature curves, are shown in Fig. 3.

Firing Procedure:

The test specimens from each type of concrete were fired at temperatures in 100 C increments up to 1000 C. Slight deviations from this procedure were made where necessary, either by omitting a certain firing temperature, or by making additional firings at intermediate temperatures. For example, no firing was carried out at 100 and 900 C on phonolite concrete, whereas on anorthosite concrete additional firings were made at 150 and 1100 C.

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[4] Methods of Fire Tests of Building Construction and Materials (E 119), 1961 Book of ASTM Standards, Part 5.

In each firing, the specimens were heated to the desired temperature and held at that temperature for 1 hr. Of the two thermocouples, the bottom one showed temperatures 100 to 250 C lower than those shown by the top one. The nominal temperature of the firing environment was assumed to be that shown by the bottom thermocouple. The top thermocouple measured the temperature of the combustion gases before they reached the test specimens. It decreased gradually during the heat-soaking period. Figure 4 shows typical temperature records.

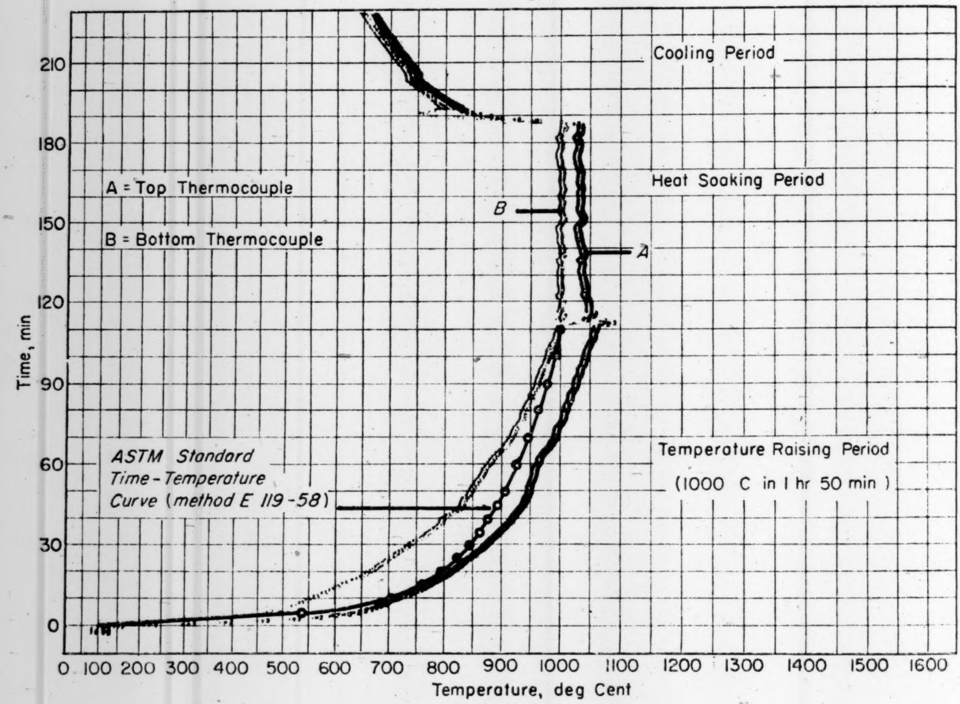


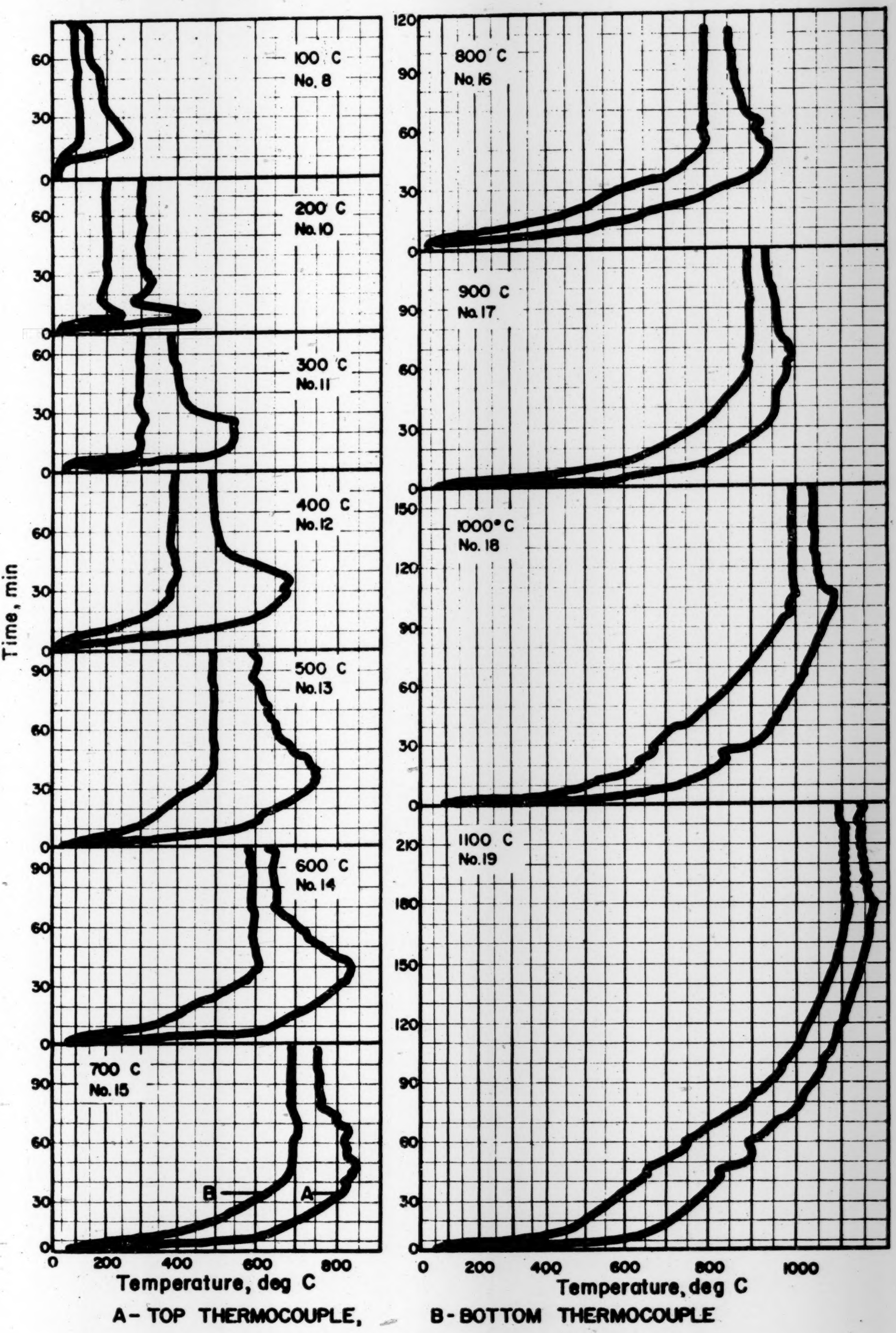
FIG. 3.—Preliminary Firing—Temperature Record.

After firing, the test specimens were left in the closed furnace to cool slowly. When they had cooled overnight to about 100 C, they were placed in a desiccator to bring them to ambient room temperature in about 4 hr and held there until tested.

Special Firing for Expanded Shale Concrete:

Before the expanded shale test specimens were fired at 1000 C, small test pieces approximately 1 by 2 by $3\frac{1}{2}$ in. were cut from an unfired reference beam and fired in a natural-gas-fired, up-draft furnace with a hearth area of 4 by 8 in., in which they could be observed during firing.

The test pieces were heated to furnace temperatures of 1000, 1070 and 1100 C at an average rate of about 14 deg per min, held at the maximum temperature for 1 hr, and allowed to cool to about 600 to 700 C before being removed from the furnace.



A, top thermocouple; *B*, bottom thermocouple.

FIG. 4.—Typical Furnace Temperature Record (Anorthosite Concrete Firing).

TABLE 10.—SUMMARY OF TEST RESULTS—EXPANDED SHALE CONCRETE.

Exposure Temperature, deg C	Weight Measurements, lb				Beam Length,[a] Dial Readings, in.		Compressive Strength, psi				Flexural Strength of Beams, psi		Pulse Velocity, ft per sec	
	Cylinders		Beams				Cylinders		Beam Ends					
	Before	After	Before	After	Before	After	Reference[b]	Fired	Reference[b]	Fired	Reference[b]	Fired	Before	After
100	5.083	5.042	11.088	11.010	0.0096	0.0105	2020	1560	2550	1850	390	315	11 500	11 100
200	5.073	4.825	10.939	10.489	0.0210	0.0203	2110	1340	2650	1720	385	240	11 200	9 980
300	5.089	4.692	10.832	10.152	0.0074	0.0020	2250	1180	2380	1460	340	190	11 100	8 960
400	5.057	4.639	10.973	10.199	0.0076	0.0016	2110	1150	2650	1510	385	150	11 300	8 770
500	5.090	4.668	10.936	10.087	0.0100	0.0011	2020	880	2550	1250	390	115	11 400	7 970
600	4.952	4.477	10.997	9.993	0.0245	−0.0069	2160	960	2460	1670	395	135	11 530	8 670
700	5.055	4.543	11.009	9.910	0.0277	...	2250	980	2380	1230	340	130	11 200	7 050
800	4.954	4.478	10.584	9.630	0.0330	0.0004	2030	820	2440	1190	385	165	10 800	7 100
900	4.966	4.448	10.846	9.779	0.0197	−0.0320	2160	550	2460	990	395	160	11 050	7 620
1000	4.987	4.520	10.846	9.789	0.0358	0.0140	2030	450	2440	550	385	137	11 300	7 290

[a] Beam gage length 14.0000 in.

[b] Reference, not-fired companion test specimens.

TABLE 11.—SUMMARY OF TEST RESULTS—ANORTHOSITE CONCRETE.

Exposure Temperature, deg C	Weight Measurements, lb				Beam Length,[a] Dial Readings, in.		Compressive Strength, psi				Flexural Strength of Beams, psi		Pulse Velocity, ft per sec	
	Cylinders		Beams				Cylinders		Beam Ends					
	Before	After	Before	After	Before	After	Reference[b]	Fired	Reference[b]	Fired	Reference[b]	Fired	Before	After
100	8.397	8.383	18.562	18.527	0.0903	0.0908	2730	2675	3570	3350	510	460	14 440	13 850
150	8.397	8.326	18.455	18.295	0.0800	0.0808	2730	2395	3570	3155	510	425	15 090	13 630
200	8.414	8.228	18.352	18.013	0.0811	0.0794	2660	1990	2640	2810	495	290	14 360	11 770
300	8.325	8.036	18.861	18.338	0.0850	0.0825	2630	1620	2790	2650	530	235	15 150	9 310
400	8.498	8.150	18.494	17.755	0.0989	0.0950	3070	1500	3525	2810	520	190	14 310	7 180
500	8.313	7.905	18.813	17.990	0.0890	0.0884	2630	1325	2790	2090	530	160	14 500	5 510
600	8.421	7.999	18.646	17.758	0.0873	0.0935	2740	1150	2775	1820	485	140	14 310	4 750
700	8.320	7.876	18.570	17.654	0.0948	0.1054	2740	1120	2775	1830	485	125	14 660	4 620
800	8.329	7.869	18.369	17.418	0.1019	0.1248	2660	830	2640	1340	495	105	14 810	3 750
900	8.416	7.960	18.486	17.514	0.1098	0.1360	2510	560	2695	1145	545	95	14 590	3 160
1000	8.434	7.964	18.491	17.511	0.0484	0.1060	2510	450	2695	645	545	85	14 750	2 540
1100	8.475	8.018	18.423	17.405	0.0829	0.1724	3070	990	3525	1210	520	110	14 370	4 140

[a] Beam gage length 14.0000 in.

[b] Reference, not-fired companion test specimens.

TABLE 12.—SUMMARY OF TEST RESULTS—ILMENITE CONCRETE.

Exposure Temperature, deg C	Weight Measurements, lb				Beam Length,[a] Dial Readings, in.		Compressive Strength, ps				Flexural Strength of Beams, psi		Pulse Velocity, ft per sec	
	Cylinders		Beams				Cylinders		Beam Ends					
	Before	After	Before	After	Before	After	Reference[b]	Fired	Reference[b]	Fired	Reference[b]	Fired	Before	After
100	13.591	13.534	29.329	29.294	0.0226	0.0256	6170	2330	7000	5085	756	538	16 150	14 250
200	13 600	13.335	29.616	29.122	0.0463	0.0476	6170	2010	7000	2850	756	355	16 400	12 300
300	13.504	13.160	30.037	29.382	0.0265	0.0271	5900	1720	7580	3210	695	285	15 600	11 500
400	13.541	13.091	30.029	29.175	0.0167	0.0137	5900	1360	7580	2340	695	180	15 820	7 490
500	13.508	13.012	29.935	28.913	0.0130	0.0073	5390	1300	7940	2130	780	145	15 720	6 820
600	13.604	13.109	29.883	28.806	0.0200	0.0168	5390	1360	7940	2370	780	145	15 900	5 360
700	13.510	12.969	29.724	28.597	0.0262	0.0262	5870	1320	7210	1950	700	90	16 400	4 620
800	...	...	...	...	...	...	...	...	...	...	...	...	...	...
900	13.697	13.183	29.926	28.777	0.0239	0.1072	6470	532	6940	1450	836	70	16 500	[c]
1000	13.711	13.179	29.609	28.402	0.0282	0.1804	6470	290	6940	790	836	40	16 200	[c]

[a] Beam gage length 14.0000 in.

[b] Reference, not-fired companion test specimens.

[c] Too damaged to be tested.

TABLE 13.—SUMMARY OF TEST RESULTS—PHONOLITE CONCRETE.

Exposure Temperature, deg C	Weight Measurements, lb				Beam Length,[a] Dial Readings, in.		Compressive Strength, psi				Flexural Strength of Beams, psi		Pulse Velocity, ft per sec	
	Cylinders		Beams				Cylinders		Beam Ends					
	Before	After	Before	After	Before	After	Reference[b]	Fired	Reference[b]	Fired	Reference[b]	Fired	Before	After
200	7.937	7.725	17.669	17.094	0.0775	0.0775	2765	1900	5060	2920	510	325	16 350	13 090
300	7.755	7.406	17.793	17.023	0.0657	0.0650	2140	970	3890	2290	545	185	15 510	8 175
400	7.942	7.439	17.789	16.649	0.0577	>0.1423	2820	220	5015	[c]	538	55	15 600	[c]
500	7.967	7.396	17.896	16.550	0.0335	>0.1665	2820	520	5015	[c]	538	60	15 875	[c]
600	7.909	7.332	17.901	16.458	0.0816	0.1161	2740	945	5520	1430	680	80	15 505	3 270
700	7.774	7.185	17.946	16.529	0.0674	0.1023	2140	640	3890	1345	545	90	15 500	3 550
800	7.820	7.177	17.697	16.239	0.0603	0.1260	2765	510	5060	1220	510	80	15 750	3 480
1000	7.980	7.293	18.064	16.396	0.0748	>0.1252	2950	1030	6000	1155	670	110	16 345	3 990

[a] Beam gage length 14.0000 in.

[b] Reference, not-fired companion test specimens.

[c] Too damaged to be tested.

These firings indicated that at a temperature of about 1070 C some underbloated shale particles started bloating, resulting in pop-outs and dilation of concrete.

Special Firing for Phonolite Concrete:

Firing of the phonolite concrete specimens at a temperature of 1000 C had indicated the tendency of the aggregate and cement to fuse together. In order to determine the rate at which fusion progresses, one special test firing was made. A test piece $1\frac{1}{2}$ by $1\frac{3}{4}$ by $3\frac{1}{2}$ in. was cut from an unfired reference beam, placed on a 1-in. refractory slab, and fired in the updraft furnace described above. The furnace was heated at an average rate of about 10 deg per min and the development of fusion was observed by opening the furnace door at intervals between 1000 and 1250 C. At 1100 C, small blisters were observed on the test piece, and at 1225 C it started to deform. At 1250 C, fusion had progressed to the extent that the test piece flowed over the sides of the slab and onto the hearth of the kiln (see Fig. 10).

TABLE 14.—THERMAL CONDUCTIVITY OF CONCRETE.

Type of Concrete Aggregate	Room-Dry Concrete Density, lb per cu ft	Thermal Conductivity		
		Mean Temperature		Coefficient *k*, Btu in. per hr sq ft deg F
		deg F	deg C	
Expanded shale	84.96	290	143	4.29
		570	299	3.28
		1051	566	3.28
		1484	807	3.42
Anorthosite	143.25	298	148	7.79
		603	317	6.86
		1132	611	6.01
		1577	858	6.14
Ilmenite	231.01	330	165	9.91
		654	345	8.26
		1195	646	7.84
		1470	800	...
Phonolite	134.90	288	142	9.60
		437	225	8.51
		662	350	6.07
		1549	843	5.04

PHYSICAL TESTS AND METHODS

Physical Tests:

To evaluate the effect of high temperatures, the following tests were carried out on the fired test specimens: (1) weight change determinations, (2) length change determinations, (3) compression tests on cylinders, (4) flexural tests on beams, (5) ultrasonic pulse velocity determinations, (6) thermal conductivity tests on slabs, and (7) petrographic examinations of the heated and broken test specimens. The test specimens were subjected to weight, length, and pulse velocity measurements before and after firing.

Methods:

Test specimens were weighed with an accuracy of ±0.001 lb.

The lengths of the beams were measured, using a 16-in. comparator designed and built at the Mines Branch. It is equipped with an Ames dial gage reading to 0.0001 in.

Concrete cylinders were tested for compressive strength according to ASTM Method C 39.[5] For all compression tests, an Amsler testing machine of 600,000 lb capacity, with a pendulum dynamometer, was used.

Concrete beams were tested for flexural strength according to ASTM Method C 78.[6] The beams were tested in a manually-operated, lever-type Tinius Olsen testing machine, of which the dial in the 10,000 lb range is graduated to 1 lb.

Ultrasonic pulse velocity was determined by a UCT electronic concrete tester.[7] This instrument operates at a 100-kc frequency with 0.1-μsec sensitivity.

Thermal conductivity was determined on 3 by 13 by $17\frac{1}{2}$ in. concrete slabs in accordance with ASTM Method C 417.[8]

The petrographic examination of the fired and broken test specimens was carried out by the Division's petrologist, using chemical analysis where necessary.

TEST RESULTS

The results of physical tests made on the four types of concrete are compiled in Tables 10 to 13. These tables show the results of weight, length, and pulse velocity measurements before and after firing. Also shown are compressive and flexural strengths of fired test specimens in comparison with strengths of companion reference test specimens.

Thermal conductivity values for the four types of concretes are shown in Table 14. For each test slab, the determination of thermal conductivity was made at four temperatures, with the mean temperature ranging from about 140 to 860 C. Due to the excessive thermal deformation and buckling of the test slab, no tests could be made on ilmenite concrete at 800 C.

Physical features of fired and broken concrete test specimens are shown in Table 15. Included also are the results of stereomicroscopic studies of the ruptured surfaces, that is, aggregate mortar bond and amount and nature of ruptured aggregate particles.

Photographs in Figs. 5 to 10 illustrate the damage sustained by different concrete test specimens after firing at various temperatures.

DISCUSSION OF RESULTS

The results obtained from tests on the fired specimens and compiled in Tables 10 to 13 are shown in Figs. 11 to 15 as percentages of either the prefired values or the values of the reference test specimens.

The weight losses of beam specimens, expressed in per cent of weight before firing, are shown in Fig. 11. The loss of weight of concrete during heating appears to have taken place in three stages, as follows:

1. *Drying*—evaporation of free moisture by heating to temperatures from 100 to 200 C.

2. *Dehydration*—loss of the nonevaporable water, or the water of hydration from the principal hydrated aluminate compounds, by heating from 200 to 300 C (**23,24**). According to Lea (**16**) the cubic hydrated aluminate compound[9] C_3AH_6

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[5] Method of Test for Compressive Strength of Molded Concrete Cylinders (C 39), 1961 Book of ASTM Standards, Part 4.

[6] Method of Test for Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading (C 78), 1961 Book of ASTM Standards, Part 4.

[7] Manufactured by A. E. Cawkell, Electronic Engineers, 6–7, Victory Arcade, The Broadway, Southall, Middlesex, England.

[8] Method of Test for Thermal Conductivity of Castable Refractories (C 417), 1961 Book of ASTM Standards, Part 5.

[9] The abbreviations used for the conventional formulas are: C_3AH_6 = $3CaO \cdot Al_2O_3 \cdot 6H_2O$ (cubic compound); AH_3 = $Al_2O_3 \cdot 3H_2O$ (alumina hydrate gel, gibbsite); CAH_{10} = $CaO \cdot Al_2O_3 \cdot 10H_2O$ (pseudohexagonal compound).

TABLE 15.—PHYSICAL FEATURES OF BROKEN HEAT-EXPOSED CONCRETE CYLINDERS AND BEAMS.

After J. A. Soles.

Type of Concrete	Temperature, deg C	Color, Appearance	Aggregate-Mortar Bond	Broken Aggregate	Conclusions
Expanded shale	Unheated	Medium dark gray.	Good, strong bond.	20 to 30 per cent on beams.	. . .
	200 to 300	Medium gray, whitened in core.	Bond appears strong.	15 to 20 per cent on beams, 5 per cent on cylinders.	Bond weakened by heating
	400 to 700	Outer $\frac{3}{4}$ in. rim brownish gray; map cracking, few larger cracks.	From strong to becoming firm, mortar slightly powdery.	10 per cent or less on beams; nil on cylinders.	Map cracking—weakening of bond, suggests shrinkage of paste.
	800 to 1000	Light pinkish gray to rusty tan; core rusty pink; more cracking.	Bond weak to very weak; mortar powdery and friable.	5 per cent or less on beams.	Color indicates oxidation of iron. Aggregate appears to be unaffected.
Anorthosite	Unheated	Dull gray.	Good, strong bond.	30 to 40 per cent on beams.	. . .
	100 to 300	Dull gray, pinkish in core.	Bond appears strong.	35 to 20 per cent on beams.	. . .
	400 to 700	Grayish pink, darker border; disintegrates along edges at 700.	Strong to fairly strong bond.	20 to 10 per cent on beams.	Slight oxidation of ferrous impurities; mortar becoming weak.
	800 to 1000	Pinkish to yellowish brown; fine map cracking at 1000.	Weak bond, mortar appears friable.	10 to 5 per cent on beams.	Mortar weak, aggregate particles appear sound.
	1100	Brownish rim, pinkish core.	Bond strong; mortar cemented.	15 to 20 per cent on beams.	Mortar sintered, "ceramic bond" started.

Ilmenite	Unheated	Medium dark gray.	Good, strong bond.	20 to 25 per cent on beams.	. . .
	100 to 300	Medium gray, whitened rim; no visible cracks.	Bond good; aggregate clean on broken cylinders.	10 per cent on beams; nil on cylinders.	Whitening suggests chemical deterioration of mortar.
	400 to 700	Medium gray to rusty pink; few map cracks.	Bond fairly strong; mortar slightly flaky to powdery.	5 per cent on beams, mostly small aggregate.	Bond weakening; flaking suggests deterioration of mortar.
	800 to 1000	Pinkish buff, rusty rim; few map cracks.	Bond appears firm to weak; mortar friable to flaky.	5 to 2 per cent, fracture along cleavages.	Concrete probably weaker than at 700; mortar very weak and crumbling.
Phonolite	Unheated	Dull gray.	Good strong bond.	30 to 50 per cent on beams.	. . .
	200	Muddy gray; darker rim.	Bond appears strong.	15 to 20 per cent on beams.	No significant change.
	300 to 600	Light brownish to pinkish gray; extensive cracking above 400.	Bond weak to very weak; at 500 largely disintegrated.	5 to 0 per cent on beams.	Phonolite unstable at 400 to 600. Later reaction with air causes decomposition.
	700 to 800	Pinkish gray interior, yellowish brown rim; abundant cracks.	Bond poor; flaky mortar; exterior more cohesive.	0 to 5 per cent; at 800 aggregate appears sound.	Mortar weakened; higher temperatures slightly improve resistance to air attack.
	1000	Rusty brown; large cracks; boils, pop-outs at surface.	Bond appears strong; mortar cemented; ringing sound.	20 to 30 per cent; most particles ruptured.	Some aggregate particles melted; incipient fusion strengthen mortar bond.

loses most of its water between 225 and 275 C.

3. *Decomposition*—dissociation of hydrous aggregate following release of the chemically bonded water, and dehydration of other hydrated aluminates, by heating from 300 to 800 C (24).

The permanent length changes for the four types of concretes after firing are shown in Fig. 12. The expanded shale concrete specimens showed gradual shrinkage up to 500 C; beyond this temperature the shrinkage increased rapidly to a maximum of 0.25 per cent at 900 C.

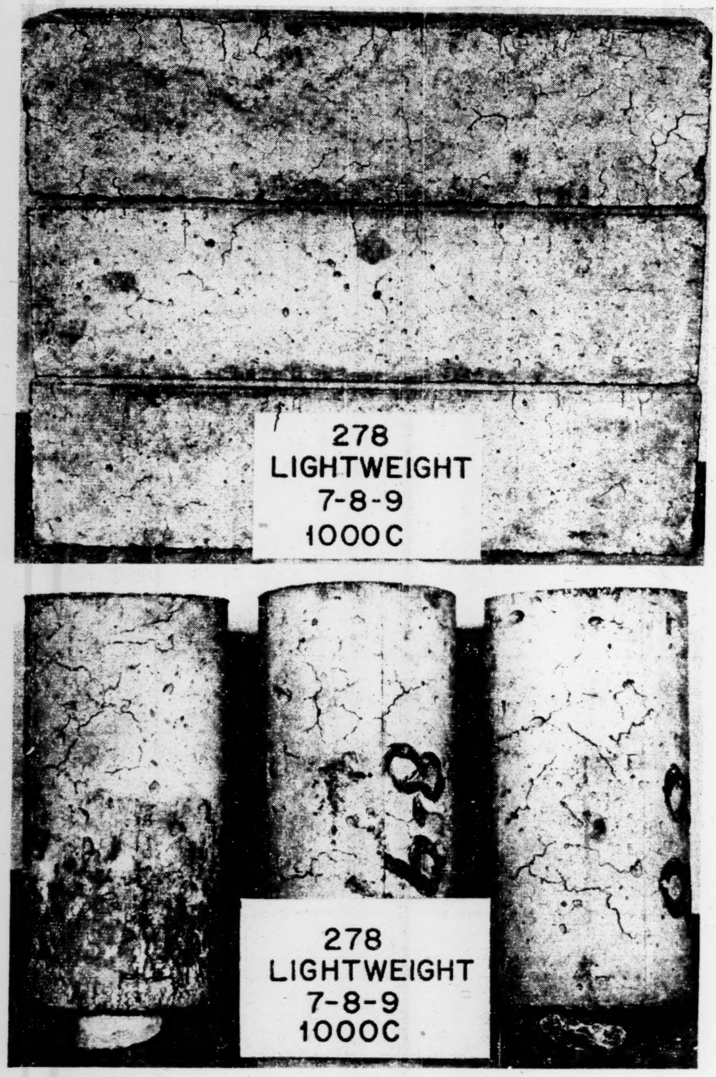
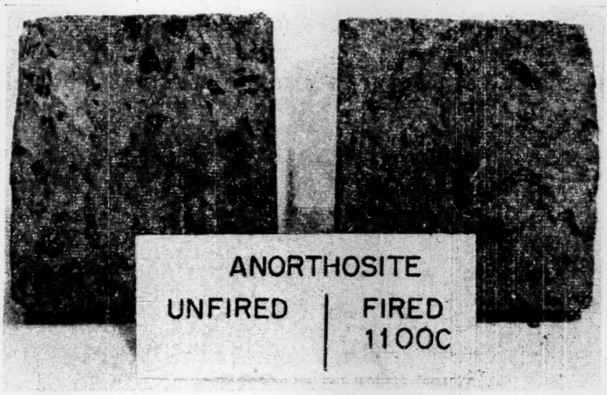
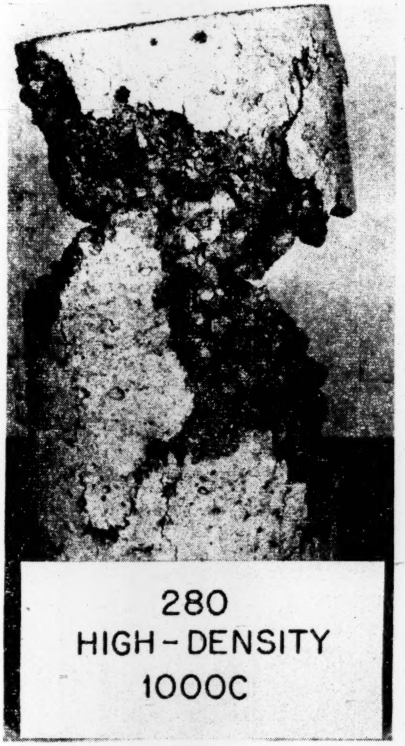


FIG. 5.—Expanded Shale Concrete Test Specimens Fired at 1000 C. Note extensive surface cracking.



(*a*) Ruptured surfaces of anorthosite concrete beams.



(*b*) Ilmenite concrete cylinder shows very poor aggregate-mortar bond. Mortar crumbled on handling.

FIG. 6.—Unfired and Fired Test Specimens After Testing.

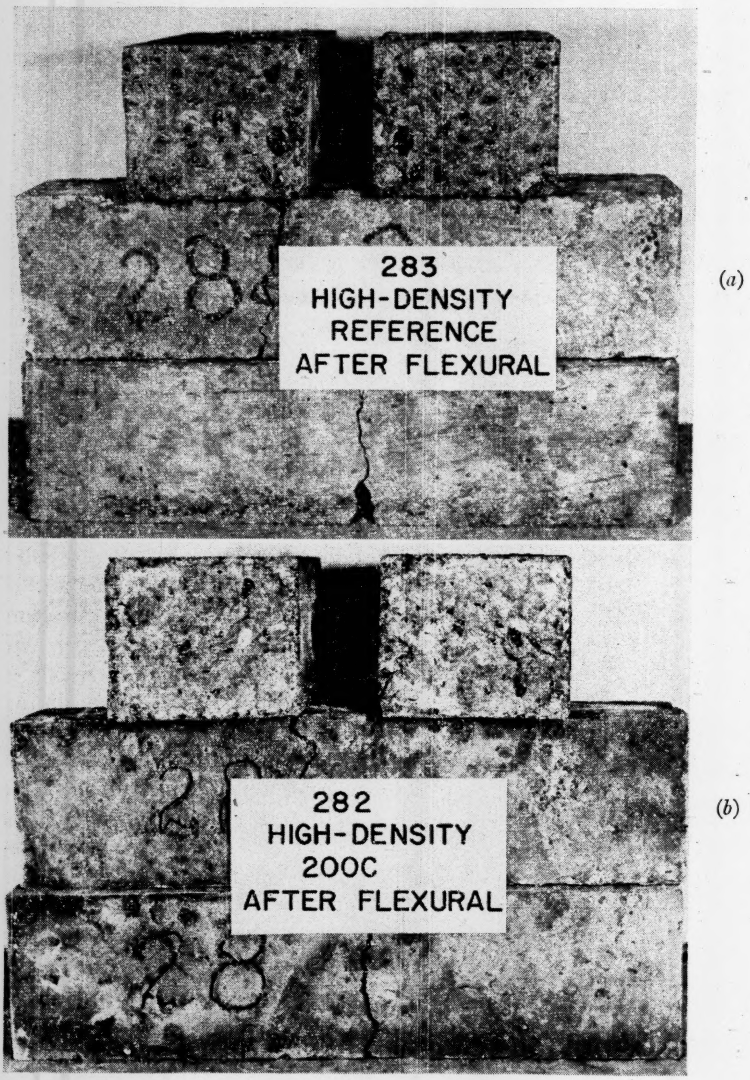
When heated to 1000 C a slight dilation of the fired test specimen was recorded, which probably could be attributed to the development of gases in underbloated shale particles.

All concretes showed a slight dilation followed by contraction, after being exposed to temperatures ranging from 100 to 500 C. A special case was phonolite concrete which suffered a sudden large expansion between 300 and 600 C. After firing at 500 C its permanent expansion was greater than 1.2 per cent; the fired test specimens were cracked extensively, indicating a decrease in aggregate-cement bond strength (see Fig. 8 (*a*)). When left in air for several days, the cracks gradually opened until the concrete disintegrated and specimens crumbled (see Fig. 8(*b*) and (*c*)). The decomposed, powdery particles of aggregate suggested a rehydration or slaking of the calcined rock under the attack of atmospheric moisture. This was substantiated by the slight weight increase of test specimens.

The expansion characteristics of phonolite concrete indicate that 300 C is a critical temperature for the phonolite (tinguaite), the temperature of dissociation of which, as determined by the thermogravimetric method, is from 300 to 450 C (**13**).

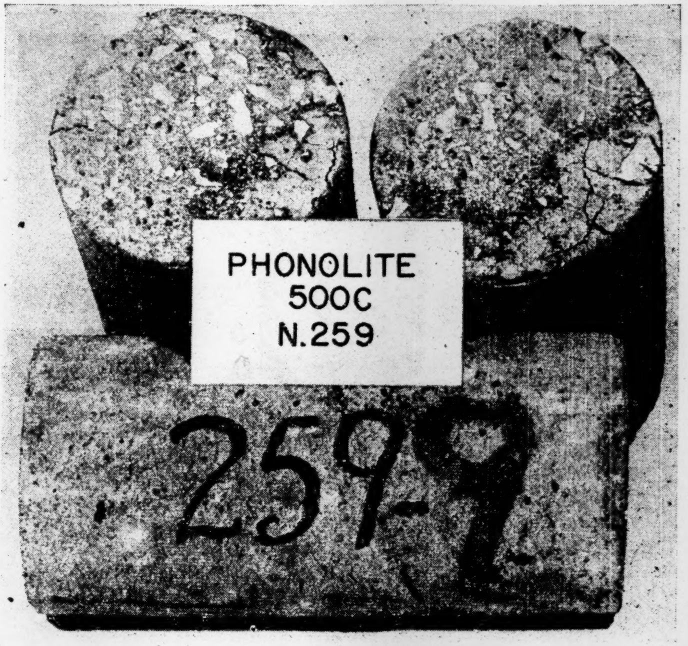
All concretes, except that made with expanded shale, showed marked expansion after being exposed to temperatures over 700 C. Of the three, ilmenite concrete showed the greatest permanent expansion, which was over 1.0 per cent after being fired at a temperature of 1000 C.

It is known from published data (6,25) that neat hydrated aluminous cement paste produces permanent shrinkage after exposure to high temperatures; when fired at 1000 C its shrinkage is about 2.0 per cent. Therefore, assuming no expanding phase is formed by cement-aggregate reaction, the resultant expan-

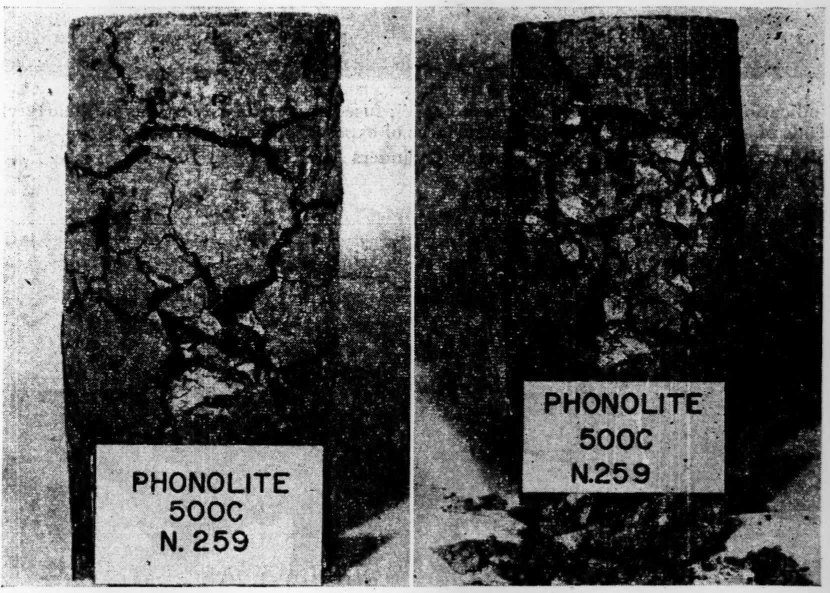

283
HIGH-DENSITY
REFERENCE
AFTER FLEXURAL
(a)
282
HIGH-DENSITY
200C
AFTER FLEXURAL
(b)

(*a*) Approximately 25 per cent of aggregate on ruptured surface broken.
(*b*) Approximately 10 per cent of aggregate on ruptured surface broken.

FIG. 7.—Unfired and Fired Ilmenite Concrete Beams After Testing.

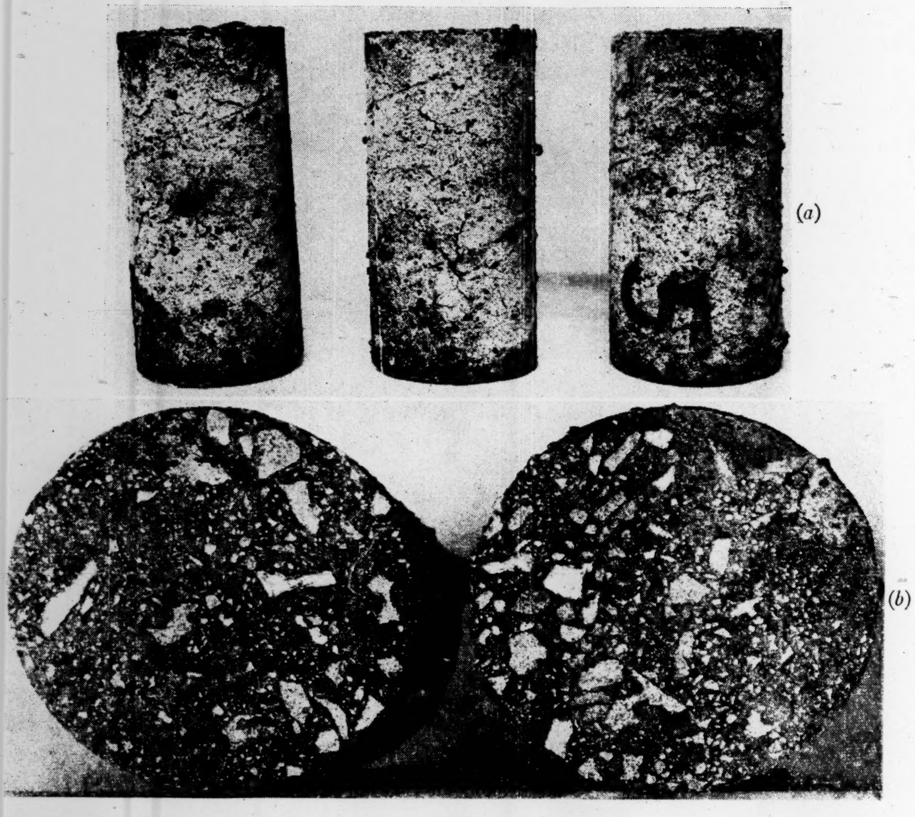

PHONOLITE
500C
N.259
259-9

(*a*) Immediately after removal from furnace; note aggregate exposed on cylinder ends.


PHONOLITE
500C
N. 259
PHONOLITE
500C
N.259

(*b*) After 7 days of air storage, at room temperature. (*c*) Same cylinder after 15 days air storage.

FIG. 8.—Phonolite Concrete Cylinders After Firing at 500 C.



(*a*) Indication of development of "ceramic bond"; fused aggregate extrustions are clearly visible.
(*b*) Ends of same cylinders. Note glossy surfaces of exposed phonolite.

FIG. 9.—Phonolite Concrete Cylinders After Firing at 1000 C.

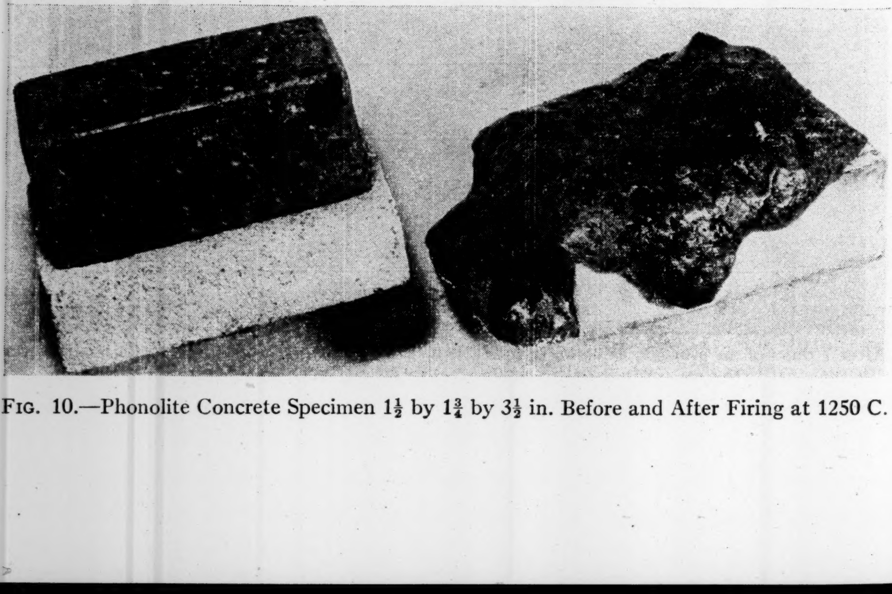


FIG. 10.—Phonolite Concrete Specimen 1½ by 1¾ by 3½ in. Before and After Firing at 1250 C.

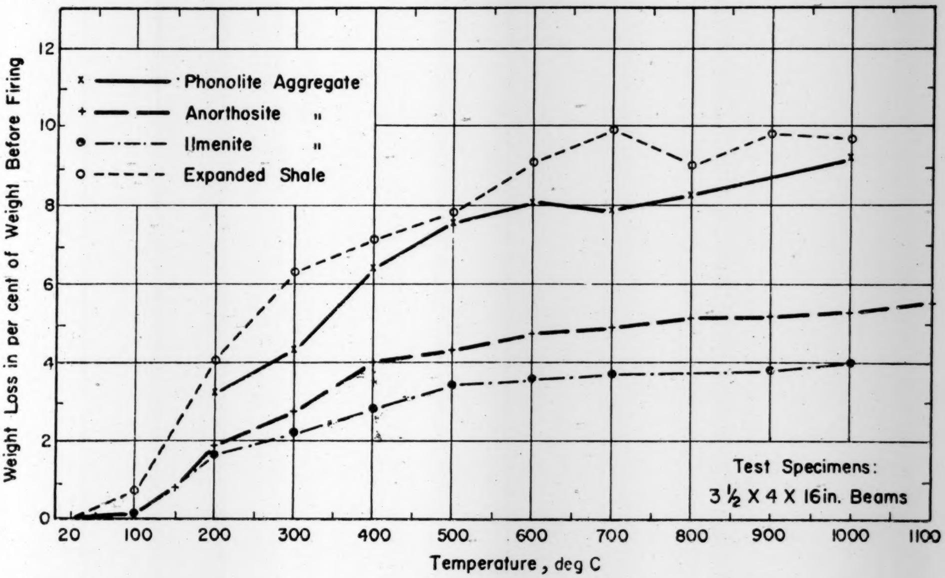


FIG. 11.—Weight Loss.

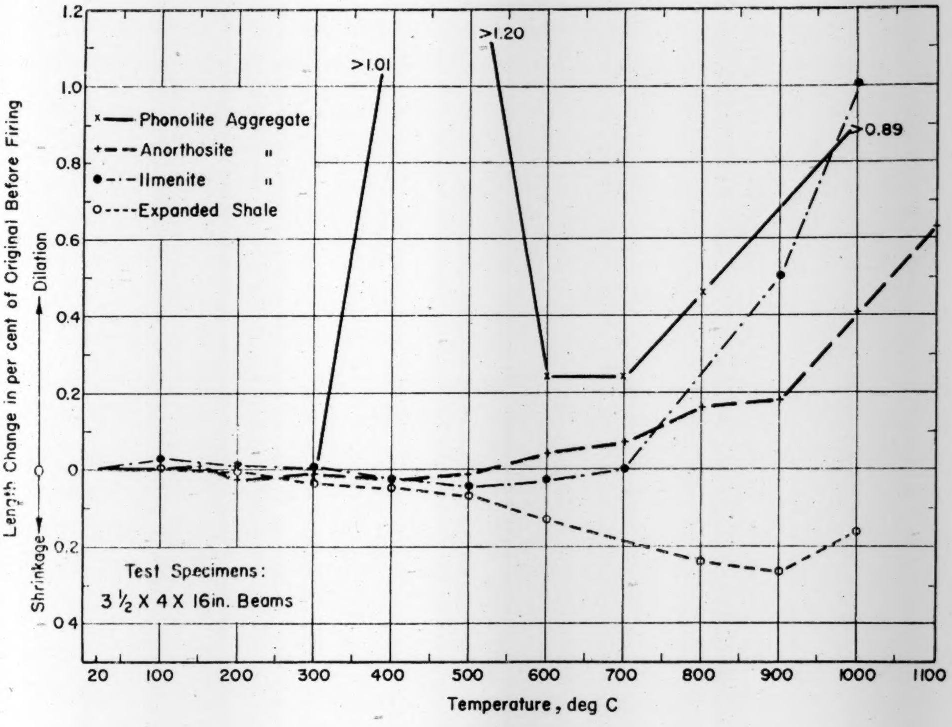


FIG. 12.—Length Change.

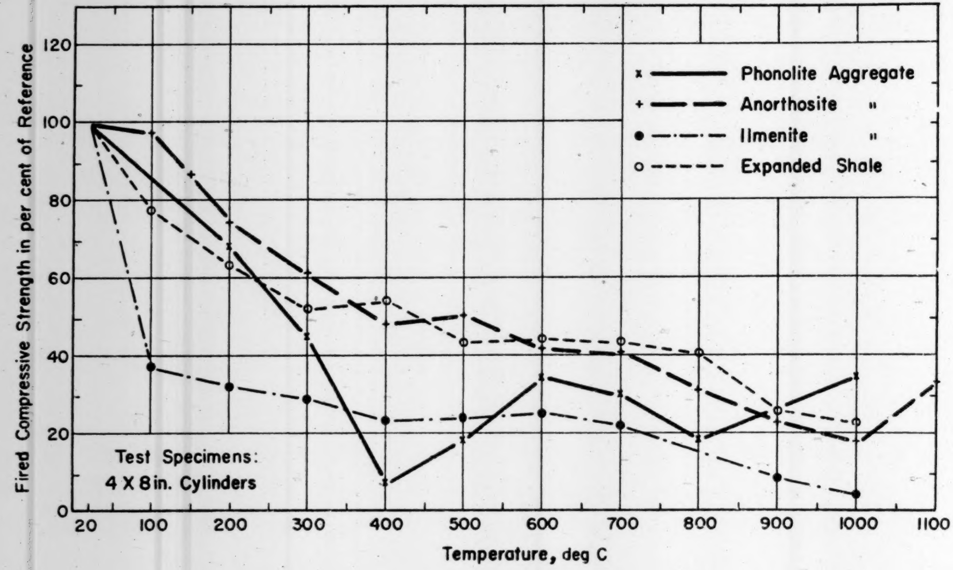


FIG. 13.—Compressive Strength.

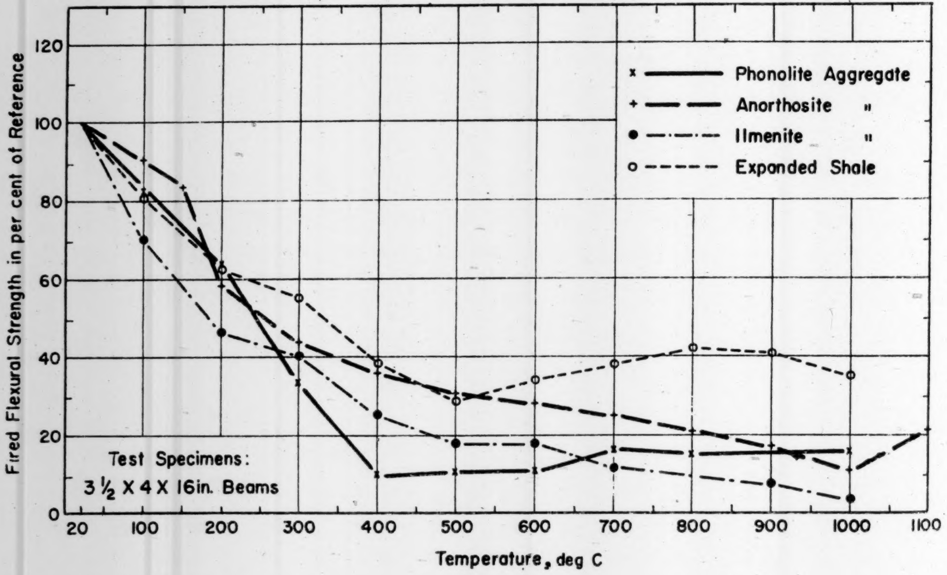


FIG. 14.—Flexural Strength.

sion of concrete exposed to temperatures over 700 C is probably caused by the expansion characteristics of the mineral constituents of the particular aggregate used.

Compressive strength changes for the four types of concretes after firing at different temperatures are shown in Fig. 13.

Concretes made with expanded shale

and anorthosite aggregates lost about half of their strength after exposure to 400 C. The compressive strengths reached a minimum of 25.8 and 17.9 per cent, respectively, after firing at 1000 C. A slight regain in strength occurred for anorthosite concrete at 1100 C.

The compressive strength of phonolite concrete specimens reached a minimum of 7.8 per cent at 400 C. Between 400 and 1000 C its strength recovered to a value of 34.9 per cent at 1000 C.

Ilmenite concrete lost more than 60 per cent of its strength after exposure to 100 C. This is comparable with relative strength losses reported by Miller and Faulkner (1) and by Arshinov (9) on aluminous cement concretes made with different aggregates. After firing at 1000 C, the residual strength of ilmenite concrete was only 4.5 per cent. This could be due to a complete breakdown of the bond between the cement paste and aggregate (see Fig 6(*b*)).

The gain in strength at 1000 and 1100 C for phonolite and anorthosite concretes, respectively, is probably due to the development of a ceramic bond. This was substantiated by visual inspection of fired test specimens and microscopic examination of the broken sections of concrete (see Table 15 and Fig. 9).

It is interesting to note that the fired strengths of concretes made with anorthosite, ilmenite, and phonolite aggregates after heating at 200 C are about the same, ranging from 1900 psi (phonolite) to 2010 psi (ilmenite). The cold strengths of these concretes were 2765 and 6170 psi, respectively. This leveling of the residual strength of concrete after heat exposure at low temperatures is largely governed by the strength of the fired cement bond, and is independent of the concrete cold strength (26). At higher temperatures, however, aluminous cement can begin to react with some aggregate, first in the solid state (for example, with expanded shale aggregate) and then when aggregate-cement fusion occurs. A ceramic bond is formed which increases the concrete strength after cooling (5,16).

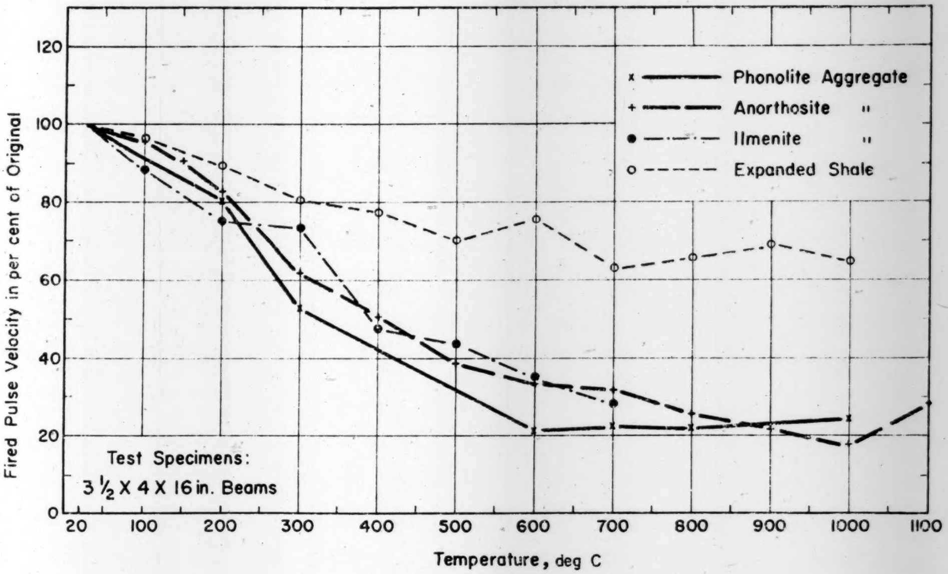


FIG. 15.—Ultrasonic Pulse Velocity.

Such a bond was formed at about 1000 C with phonolite aggregate.

Changes in flexural strength of beams of the four types of concretes after firing are shown in Fig. 14. The flexural strengths dropped rapidly upon exposure to temperatures up to 500 C. At this temperature the fired strength ranged from about 10 per cent of the original for phonolite concrete to about 30 per cent for expanded shale and anorthosite concretes. Between 500 and 1000 C, the residual flexural strength for anorthosite and ilmenite concretes decreased gradually, reaching a minimum of about 5 per cent for ilmenite concrete at 1000 C. The expanded shale concrete beams, on the contrary, showed a strength recovery, reaching about 35 per cent at 1000 C. The anorthosite concrete showed a slight regain in flexural strength at 1100 C, as it had in compressive strength.

The ultrasonic pulse velocities of the four types of concretes fired at different temperatures are shown in Fig. 15.

The pulse velocities of expanded shale concrete specimens showed a gradual drop with increased temperature, reaching a minimum value of 64.5 per cent at 1000 C.

The pulse velocities of the other concretes dropped to less than 40 per cent with firing temperatures increasing up to 600 C. Between 600 and 1000 C the pulse velocities of anorthosite concrete specimens decreased continuously, reaching a minimum of 28.8 per cent at 1000 C; the phonolite concrete specimens showed a slight recovery of pulse velocity values, reaching 24.4 per cent at 1000 C. This is probably due to the development of a ceramic bond which causes closure of microcracks developed in the interior of the concrete specimens.

It is believed that the reduction in the residual compressive and flexural strengths of aluminous cement concretes at lower ranges of elevated temperatures is related primarily to changes in the principal compound composition of the cement, although the type of aggregate also affects the strength of fired concrete greatly.

According to Schneider (25), the major decrease in the strengths of aluminous cement between 110 and 300 to 500 C is due to the dehydration of its principal compounds[9] C_3AH_6, AH_3, and CAH_{10} and the formation of their dehydration products. The changes in strength noted at temperatures from 300 to 500 C cannot be related to the formation or the presence of any one compound. Above 700 C the loss in strength was possibly due to grain growth and the formation of new compounds.

Wells and Carlson (27) suggested that the loss in strength at elevated temperatures was due to crystallization of AH_3 from alumina gel.

No measurements were made to determine Young's modulus or the dynamic modulus of elasticity of the fired test specimens. It has been suggested (7,24, 25) that changes in the strength of fired concrete are accompanied by similar, although not always proportional, changes in the elastic moduli.

The values compiled in Table 14 show the effect of different aggregates on the thermal conductivity of concrete. Of the four types of concretes tested, ilmenite had the highest thermal conductivity, closely followed by phonolite and anorthosite concretes.

The *k* values for expanded shale concrete agree closely with the graph established by Hansen and Livovich (28) for the relationship between thermal conductivity (at a mean temperature of 1000 F) and fired unit weight of insulating concretes made with aluminous cement.

The *k* factor decreases with decreasing free moisture content in concrete. Ruh and Renkey showed (29) that thermal conductivity decreases still further at temperatures ranging from 200 to 800 C, when the chemically combined water is driven off.

In this study the decreasing *k* values accompanying increasing mean temperatures indicate gradual weakening of the aggregate-mortar bond and a general deterioration of fired concrete. An increase in thermal conductivity in certain cases at the higher temperatures probably indicates the development of ceramic bond (**29**).

SUMMARY

It is concluded from this study that aluminous cement concretes on exposure to dry heat deteriorate rapidly. Extended deterioration of concrete is dependent to a large degree on the type of the aggregate.

Concretes made with expanded shale, anorthosite, ilmenite, or phonolite aggregate lose more than 50 per cent of their strength after exposure to 400 C. At temperatures between 400 and 1000 C the loss in strength continues, but at a relatively slower rate.

Expanded shale concrete showed the greatest stability, whereas phonolite concrete was the least heat-resistant of the four types investigated.

The reduction in strength of the four concretes after exposure to temperatures up to 300 C was caused, to a large degree, by a reduced residual strength of the fired mortar which, it is believed, is primarily due to changes in the principal compound composition of the cement. Between 300 and 1000 C the deterioration is governed mainly by the physical properties and mineral constituents of the aggregate.

The results of this study should be considered when designing aluminous cement concrete structures exposed to dry heat, and investigation of the concrete under the anticipated exposure conditions is recommended.

Acknowledgments:

Grateful acknowledgment is made to the following staff members of the Mineral Processing Division, Mines Branch: J. A. Soles, for carrying out the petrographic examination of the aggregates and the fired test specimens; J. G. Brady, for providing facilities for test firings and thermal conductivity tests on the concrete slabs; and F. E. Hanes, for taking numerous photographs during this investigation.

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DISCUSSION

MR. R. E. PHILLEO.[1]—Were any measurements of length and weight changes on neat cement paste specimens made in order to determine what the cement would do without the complicating effects of aggregate?

MR. N. G. ZOLDNERS (*author*).—No measurements of length and weight changes of neat aluminous cement paste were made during this investigation. However, data are available on expansion and contraction of neat aluminous cement paste showing that after a small initial expansion up to 100 C the paste begins to shrink due to elimination of combined water, and at 700 to 800 C there is further contraction due to changes in the alumina gel and hydrate structure. After cooling there is a net contraction of about 2 per cent. The residual length changes reported in the paper are the resultant of those shown by the aggregate and the aluminous cement paste.

MRS. KATHARINE MATHER.[2]—Please compare the temperatures at which castable refractories are used with those at which the authors' tests were made.

MR. ZOLDNERS.—Castable refractories are used up to 1850 C, while our tests ran up to 1000 C.

MR. T. D. ROBSON[3] (*presented in written form*).—The authors conclude by saying that their results should be considered when designing aluminous cement concrete structures exposed to dry heat, and investigation of the concrete under the anticipated exposure conditions is recommended. Their paper has therefore been examined to see whether the results are sufficiently related to practical work to provide new and valid information in this field.

Of the four aggregates employed, three are naturally occurring and the fourth is a manufactured expanded shale. The latter type of aggregate has been widely used in aluminous cement concretes for widely varied applications involving hot-face temperatures up to 1000 to 1100 C, and much information is already available on the properties and service behavior of these concretes. The suitability of this type of aggregate for its recommended duties is therefore not in doubt, but it would perhaps have been useful to test expanded shale from this particular source against expanded shales from other sources in order to compare properties in detail, or to guard against the somewhat unlikely possibility that it possessed completely unsuitable and anomalous properties.

In the case of naturally occurring aggregates, their variable nature makes it essential to determine first whether they are fundamentally suitable for use in concretes at elevated temperatures before proceeding to detailed examination of the physical properties of the fired concretes. The practical effects of excessive thermal expansion or volume change can best be revealed by preliminary heat-cycling tests, and the production of free lime or other expansive material can be discovered readily by firing-and-quenching tests. The work described by the authors is not designed for these functions but is probably sufficient to suggest that the phonolite aggregate is unsuitable for the temperature range covered satisfactorily by many other traprock aggregates, and that the service behavior of ilmenite concrete could be doubtful above 800 C. Anorthosite is not a particularly suitable aggregate of the traprock type since it is very coarse-grained, but it does not appear to present many other undesirable features; experience shows that it can give satisfactory service up to 1000 C in districts where its availability and cheapness form the main attractions.

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[1] Office of the Chief of Engineers, Headquarters, Dept. of the Army, Washington, D. C.

[2] Corps of Engineers, U.S. Army Engineer Waterways Experiment Station, Jackson, Miss.

[3] Lafarge Aluminous Cement Co. Ltd., London, England.

It is rather difficult to determine the underlying plan of the work reported. The use of the same water-cement ratio, the same ratio of coarse to fine aggregate, and the same cement content would appear to be justified only if the various types of concrete are being directly compared one with the other. But this surely cannot have been the object. The aggregates vary widely in physical characteristics and will produce concretes that are used in practice for quite different reasons; the most important property required of the fired concrete is therefore not the same in all cases. For the results of this work to have any practical value it would clearly be necessary to make each type of concrete with a mix capable of yielding the kind of properties desired in the applications for which it would normally be used. This would entail very different cement and water contents in the concretes.

A loss in strength on firing heat-resistant or refractory concrete should not be a matter for surprise, since it is an inevitable consequence of the dehydration of any hydraulic cement, but the extent of such loss is determined by many factors other than those mentioned by the authors. The user is not interested in the percentage loss but only in the maintainable residual strength; the latter, to a considerable degree, is within his own control. For example, insulation value may be the chief reason for employing an expanded-shale concrete at high temperatures, and in such cases, where no substantial fired strength is usually necessary, the use of lean mixes of high water-cement ratio may be permissible and will give the maximum value of the property desired. On the other hand, if the application necessitates the maximum fired strength obtainable from concrete made with this aggregate, then a rich mix and a low water-cement ratio will be used.

The dense-aggregate concretes would always be used for applications in which a reasonably high fired strength is likely to be required and it would be quite inappropriate to use the mix proportions or water contents reported in this paper. Unfortunately, therefore, the given fired strength results have little absolute value for general design purposes, and their utility is further reduced by the short firing time of 1 hr at the various temperatures. With this size of specimen, it is impossible for the concrete to approach either thermal or desiccation equilibrium in the time allowed, particularly at the lower temperatures.

The general impression left on the reader, and perhaps in the minds of the authors, could easily be one of doubt about the practical utility and performance of heat-resistant concretes. On the basis of practical experience this would be quite unjustified, and it has therefore been necessary to draw attention to certain basic principles which, it is hoped, may be helpful in planning future work.

MESSRS. N. G. ZOLDNERS, V. M. MALHOTRA, AND H. S. WILSON (*authors' closure by letter*).—Mr. Robson questions the underlying plan of the work reported. The plan of the investigation was to determine the effect of heat on aluminous cement concretes. The results showed that irrespective of the aggregate used, the application of aluminous cement for concretes to be exposed to heat below that at which a ceramic bond is developed is open to question.

The use of anorthosite and phonolite aggregates was requested by commercial producers and industrial users. Ilmenite was used because of its possible application in nuclear shielding concretes. The mix design for concretes was kept constant to reduce the number of variables to a minimum.

The authors agree with Mr. Robson regarding the limitations imposed by the short duration of the firing time and plan to conduct future investigations at longer firing times.

Perhaps the fired strength results given in the paper cannot directly be used for design purposes. However, the authors consider their study amply justified in that it brought out, in some measure, the extent of the strength losses suffered by the various concretes exposed to heat up to 1000 C.

[For additional discussion of this paper, see current issues of *Materials Research & Standards*.—ED.]

DETERMINATION OF MOISTURE CONTENT OF HARDENED CONCRETE BY ITS DIELECTRIC PROPERTIES

By J. R. Bell,[1] G. A. Leonards,[2] and W. L. Dolch[2]

Synopsis

Capacitances and dissipation factors for 3-in. diameter thin disks of concrete and mortar were measured in the laboratory at various water contents using conventional bridge techniques at 10^6 cps. From these data relative dielectric constants and dielectric conductivities were calculated. Capacitances were determined also at 60 and 6×10^5 cps. Conductivities at various water contents were determined at 10 cps using small prisms of concrete. The experimental variables involved and the usefulness of the dielectric properties as moisture indicators are discussed. Except for unusually severe drying of the concrete or salt content of the pore water, these properties indicated moisture contents below about 6 per cent with an accuracy of ±0.25 per cent, or for the low-frequency conductivity measurements, ±0.5 per cent.

The investigation has shown that the dielectric properties of concrete are potentially satisfactory indicators of moisture content. Successful field use of the method, however, is contingent upon a judicious selection of appropriate details of instrumentation and on careful calibration.

For many purposes, mostly in connection with problems of shrinkage and thermal conductivity, it is desirable to know the moisture content of hardened portland-cement concrete. A satisfactory method for the determination of moisture content should be nondestructive, accurate, precise, and have a rapid enough response to be useful for transient conditions.

Many methods have been tried for this purpose. Some involve destruction or disruption of the sample. Others use a relationship between the moisture content of the concrete and that of some embedded material the properties of which are determined. Radioactive meters have been used but are not yet localized enough in their region of influence for accurate measurement of moisture gradients in pavement slabs.

Theoretical considerations by Harr and Leonards (**1,2**)[3] indicated that it is desirable, with respect to slab warping studies, to measure the water content with sufficient accuracy to indicate length changes resulting from moisture variations that are equivalent to those of a temperature change of about 5 F. Various studies of shrinkage and of thermal expansion (**3,4**) show that a sensitivity of about 0.25 per cent reasonably fulfills this objective.

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[1] Associate Professor, Civil Engineering Department, Oregon State University, Corvallis, Ore.

[2] Professor and Associate Professor, respectively, Civil Engineering School, Purdue University, Lafayette, Ind.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper.

All methods previously investigated were considered to have shortcomings; however, the capacitance or dielectric constant method appeared to show the greatest potential. This method is based on the relatively large difference in dielectric constants of water (about 80) and most inorganic solids (about 5 to 10).

Two assumptions are implied in the dielectric method of measuring moisture contents: (1) that the capacitance of the sensing capacitor can conveniently be measured so as to reflect the true dielectric constant of the material being studied, and (2) that the dielectric constant - water content relationship is constant for the conditions of the investigations and can be determined by some convenient calibration procedure. To evaluate the potential of this method, the validity of these assumptions must be established for the conditions of the particular situation.

DIELECTRIC PROPERTIES OF MATTER

Using the notation of von Hippel (5), the following are brief definitions of the important variables of concern in this study.

If a capacitor of plate area A and separation d contains no matter between its plates, its capacitance C_0 will be

$$C_0 = \frac{A\epsilon_0}{d} K \ldots\ldots\ldots\ldots (1)$$

where ϵ_0 is the real permittivity (dielectric constant) of a vacuum and K is a constant depending on the units of the other terms. If material with a real permittivity of ϵ' is inserted between the plates, the capacitance will increase to

$$C = \frac{C_0\epsilon'}{\epsilon_0} = C_0\kappa' \ldots\ldots\ldots (2)$$

where κ' is the relative dielectric constant.

If a capacitor is "leaky," that is, if there is some energy dissipation mechanism inherent to it, there will be a loss current, I_l, that lags the charging current, I_c, by a phase angle of 90 deg. The vector sum of these currents is the total current through the capacitor and is separated from the charging current by the so-called loss angle, δ. The dissipation factor, D, is defined as

$$D = \tan\,\delta = \frac{I_l}{I_c} \ldots\ldots\ldots\ldots (3)$$

Since the loss current can arise from processes other than the migration of charge carriers, a complex permittivity, ϵ^*, is defined:

$$\epsilon^* = \epsilon' - i\epsilon'' \ldots\ldots\ldots\ldots (4)$$

where ϵ'' is called the loss factor. In these terms the dissipation factor is

$$D = \tan\,\delta = \frac{\epsilon''}{\epsilon'} \ldots\ldots\ldots\ldots (5)$$

and the product of angular frequency and loss factor is the dielectric conductivity,

$$\sigma = \omega\epsilon'' \ldots\ldots\ldots\ldots (6)$$

The conductivity can be caused by the movement of charged bodies such as ions, the orientation of dipoles, or any other process of energy dissipation.

A dielectric material increases the storage capacity of a capacitor by neutralizing charges at the electrode surfaces. This neutralization can be visualized as the result of the orientation or creation of dipoles to oppose the applied field. Such a polarization is proportional to the ease with which the dielectric can be polarized or its polarizability. Polarizability is the average induced polarization per unit field strength. The greater the polarizability of a material the greater will be its dielectric constant.

Four mechanisms of polarization are recognized: (1) electronic, (2) atomic,

(3) orientation, and (4) space charge or interfacial. The first three polarization mechanisms are forms of the dipole polarization previously mentioned. They result from the presence of permanent dipoles or dipoles induced by external fields. The fourth type—space charge polarization—results from charge carriers that are usually present and more or less free to move through the dielectric. When such carriers are impeded in their movement and become trapped in the material or at interfaces, space charge concentrations result in a field distortion that increases the capacitance.

These considerations indicate that the dielectric "constant" of a material cannot be constant because it is a function of the polarizability which is in turn a function of frequency, temperature, local fields, applied field strength, availability and freedom of charge carriers within the dielectric, and local field distortions.

The dielectric conductivity will also show the influence of these same factors. This is true because dielectric conductivity is a function not only of ohmic conductivity but also of the power consumed in polarizing the material; therefore it must reflect the control these factors have over the polarizability.

PREVIOUS WORK

The measurement of moisture content by means of capacitance has been done on a wide variety of materials. Some investigators of this method have expressed complete satisfaction, while others have had difficulty, which frequently has been blamed on experimental factors. Much work on soils (6–13) has shown the variation attributable to the frequency of the test signal and to certain troublesome experimental factors such as the contact between the system and the electrodes.

The same general conclusions have been reached by workers using rocks (14,15).

Anomalously high dielectric constants have been measured; in the case of moist rock specimens values higher than the dielectric constants of any of the constituent materials of the rocks tested were frequently encountered. These anomalously high values can be explained on the basis of an interfacial polarization phenomenon (15).

A paper by Hammond and Robson (16) contains the only data on the dielectric properties of concrete that could be found. These workers found anomalously high dielectric constants for frequencies in the range of 50 to 25,000 cps. They also obtained somewhat different values for different types of cement, and the dielectric constant values for pastes were significantly higher than those for concrete. All that can be learned about the dielectric constant - water content relationship from these data is that, within the range of the relatively low water contents tested, the dielectric constant generally increases with increased water content.

The conductivity of concretes and pastes has been studied by several investigators, and it is generally accepted that this property is strongly influenced by factors other than water content. Spencer (18), for example, found that at the same water content the resistivity of a concrete may vary by 40 per cent with changes in the salt content of the pore fluid.

It was decided to investigate the dielectric method for its applicability to the determination of moisture in concrete. To delineate the various important factors more fully was the main purpose of this work.

EXPERIMENTAL WORK

Materials and Samples:

The samples tested were prepared from mortars or concretes that were proportioned as follows: mortars—1:2.5:0.67 by weight of cement, fine aggregate, and water; concrete—1:2:1.75: and 0.67 by weight of cement, fine aggregate, coarse aggregate, and water. The cement was a type I portland and the aggregate was a dolomitic limestone. Both fine and coarse aggregate for the concrete were limestone for ease in specimen preparation. Some mortars were made with the stone sand and some with Ottawa sand. The maximum coarse aggregate size was ½ in.

It will be noted that the water-cement ratio was higher than that used in normal highway construction. This was done deliberately to extend the range of evaporable water investigated.

The mortars or concretes were cast in standard, 3-in. cylinder molds, cured in the molds under wet cloths for 2 days, taken from the mold, and immersed in a 100 F water bath for 5 days. Disks approximately ⅜ in. thick were then sawed from the cylinders and finished by the usual grinding and lapping techniques. All cutting and grinding was done wet. The final results were disks of two thicknesses—about 4 mm and about 7 mm. Only disks with plane and parallel sides and no noticeable irregularities were used for the permittivity measurements.

For the resistance measurements, small prisms, about ⅜ by 1 by 2 in., were cut from the rough disks and then ground and lapped.

All prepared specimens were returned to water storage, and testing was begun after 28 days.

Capacitance Tests:

The capacitance tests were run by placing the specimen, surface dry at first, in a thermostated chamber (30 C) between 3-in. diameter electrode plates insulated with a thin polytetrafluoroethylene (Teflon) sheet. The test cell was incorporated in a conventional capacitance bridge circuit and the relative capacitance determined in the usual way. The specimens were then removed from the test cell and partly dried, either at ambient or slightly elevated temperatures, and a new determination of relative capacitance made at a new, and lower, water content. On the wetting half of the cycle the specimen was exposed to water vapor or briefly immersed in water between measurements.

In such a way several cycles of wetting and drying were obtained. The driest condition for some specimens was oven-drying at 105 C, for others, drying to 60 C. In all cases the moisture content refers to comparison with the oven-dry weight at the time of the test.

Several test instruments were used. The first was a General Radio Type 740 a-c bridge operating at 60 cps. When the need to operate at higher frequencies became evident, a 6×10^5 cps bridge was made in the laboratory and used. Neither of these instruments measured dissipation factor. They gave data only for apparent capacitance, and the real dielectric constant could not be calculated from the results. Therefore a General Radio Type 1610-AH assembly was obtained which operated at 10^6 cps and all the necessary data mentioned above could be secured. The apparent capacitance and dissipation factor were determined, and the relative dielectric constant, ϵ'/ϵ_0, and dielectric conductivity, $\omega\epsilon''$, were calculated from the

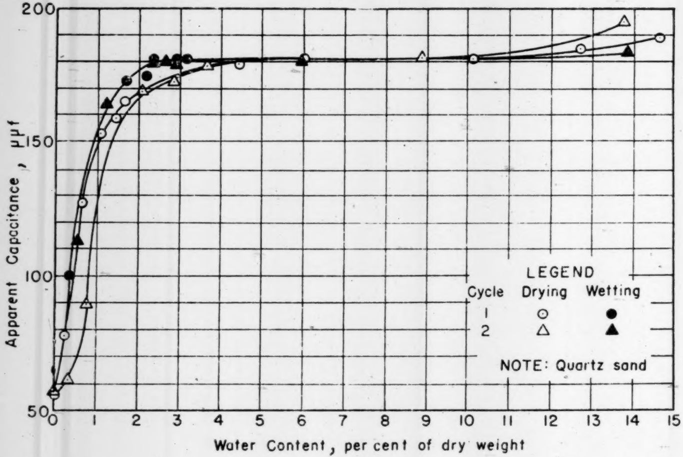

Fig. 1.—Capacitance Versus Water Content for Disk 8 at 60 cps.

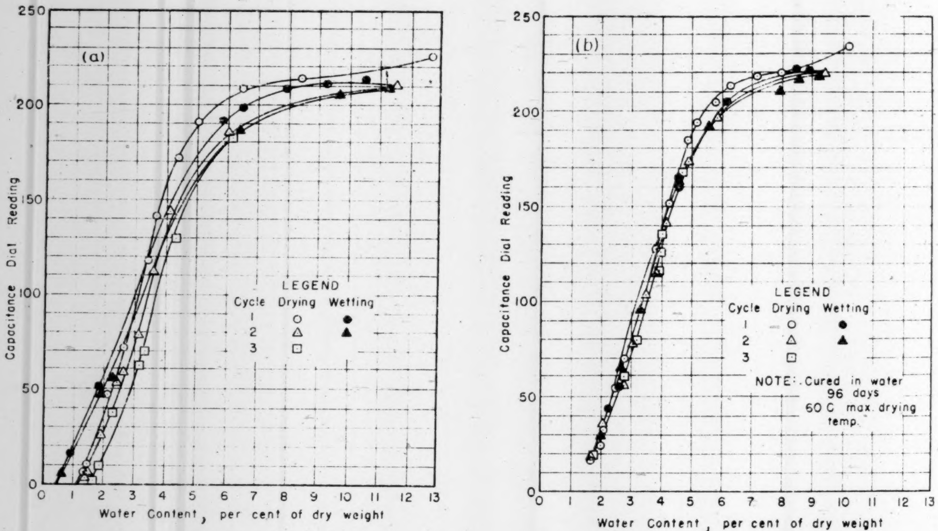

Fig. 2.—Capacitance Dial Reading Versus Water Content for (*a*) Disk B-C and (*b*) Disk C-C a 600,000 cps.

measured values by conventional methods (**18**).

Conductivity Tests:

Low-frequency conductivity tests were performed on the prisms by the use of a specially built 10-cps resistance bridge. The electrodes for these tests were prepared by painting the specimen ends with silver conducting paint.

Weighings for moisture content were, in all instances, made to 1 mg. The moisture contents were corrected to account for the increased dry weight due to continued hydration of the cement.

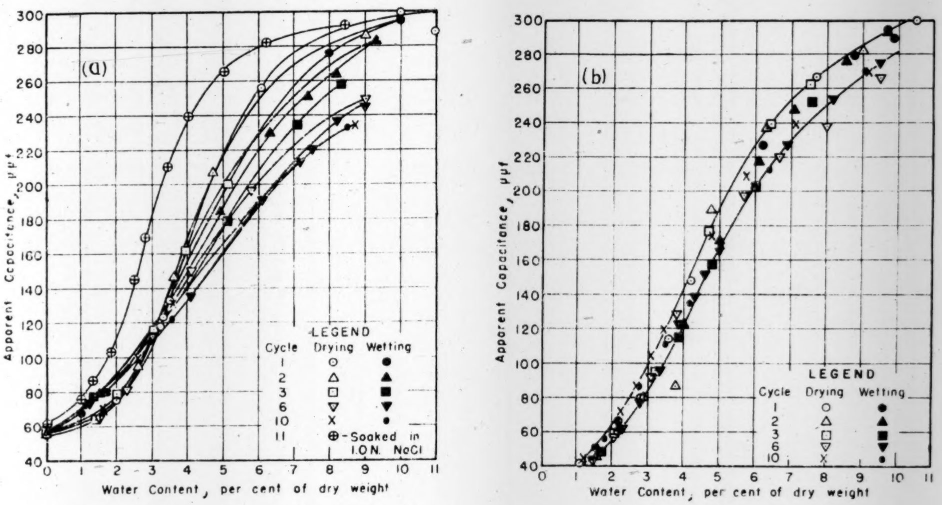

FIG. 3.—Apparent Capacitance at 10^6 cps Versus Water Content for (*a*) Disk C-1 and (*b*) Disk CT-2.

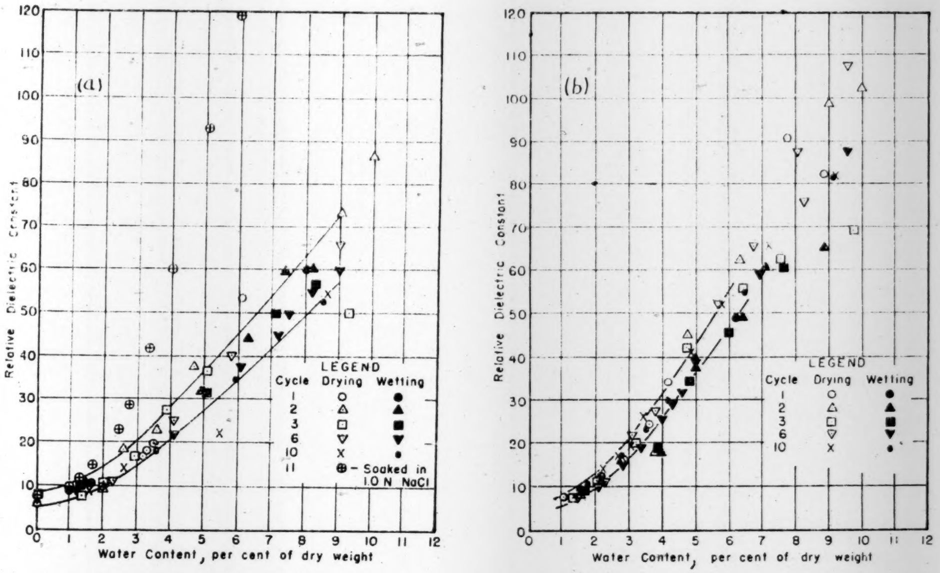

FIG. 4.—Relative Dielectric Constant at 10^6 cps Versus Water Content for (*a*) Disk C-1 and (*b*) Disk CT-2.

Data:

Only typical results are presented. Other results exhibited no features not shown here and are available elsewhere (**18**).

Figure 1 shows the results obtained in an early test on a quartz-sand mortar using the 60-cycle bridge. The values plotted are apparent capacitance and include the influence of the contact

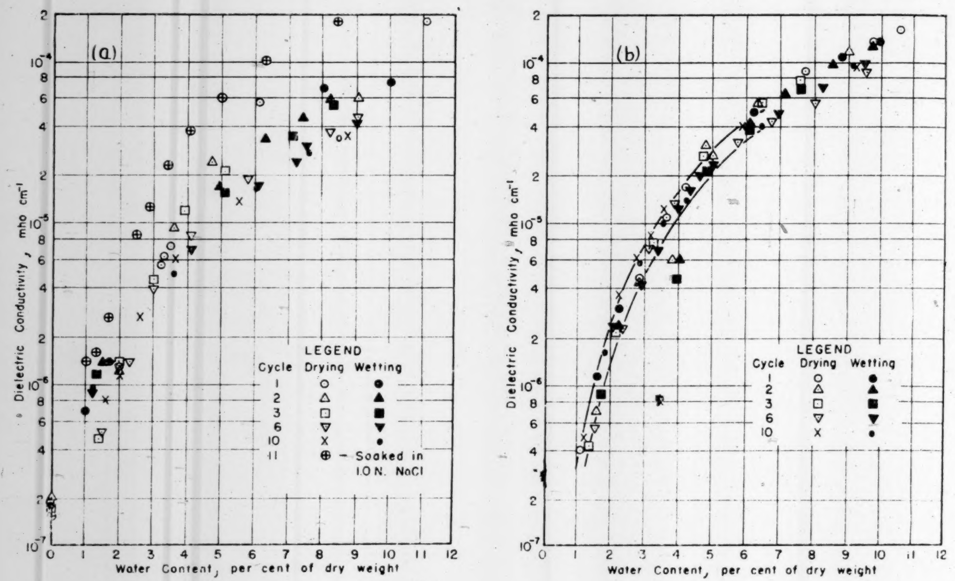

FIG. 5.—Dielectric Conductivity at 10^6 cps Versus Water Content for (*a*) Disk C-1 and (*b*) Disk CT-2.

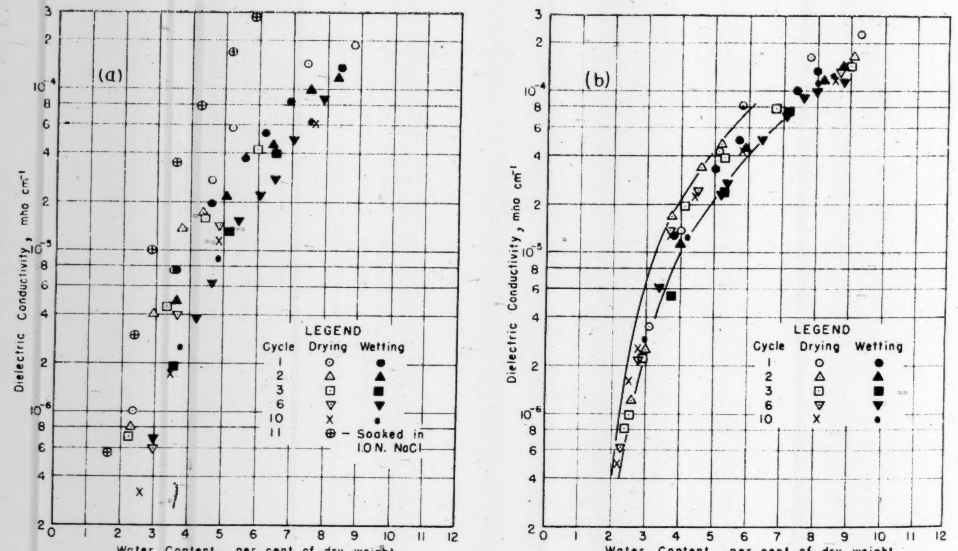

FIG. 6.—Dielectric Conductivity at 10 cps Versus Water Content for (*a*) Sample RC-1 and (*b*) Sample RC-2.

capacitance as well as the capacitance of the mortar disk itself.

Owing chiefly to the obvious insensitivity of these measurements at any but low water contents, the test series at 6 × 10^5 cycles was carried out. Data typical of these results are shown in Figs. 2(*a*) and (*b*). The specimens represented here were stone-sand mortars. Specimen C-C was dried to 105 C and specimen B-C was cured longer than usual and dried to only 60 C.

The impedance bridge used for this series was not calibrated to read capacitance directly. Therefore, the values plotted are not capacitance but only dial readings that indicate relative capacitance. These readings are a function of the contact capacitance of the test capacitor and the capacitance of the mortar disk; therefore they cannot be compared directly with the values from other test series. Only relative comparisons can be made.

Most of the tests were carried out at 10^6 cycles with the equipment that permitted the determination of both relative dielectric constant and loss factor or dielectric conductivity. For these tests, in addition to the variables previously discussed, specimens of two thicknesses were tested.

The results of these tests for two concrete disks, C-1 and CT-2, are shown in Figs. 3 to 5. These results are typical of those from this test series. The results for the mortar specimens had different absolute values, but the trends were the same. Disk C-1 was a thin (4-mm) disk dried to 105 C and disk CT-2 was a thicker (7-mm) disk dried to 60 C. These two conditions represent the extremes of the test variables. Both disks were tested through ten cycles of drying and wetting with water. Disk C-1 was also soaked in a 1.0*N* sodium chloride solution after the tenth drying and was tested through an additional drying phase.

The apparent capacitances contain the effects of both the contact and disk capacitances and are comparable to the results obtained at 60 and 600,000 cps. It was possible also to calculate the relative dielectric constants and dielectric conductivities for the conditions of these tests.

The results of the resistivity measurements at 10 cps are given for two typical concrete specimens in Figs. 6(*a*) and (*b*). Dielectric conductivity was calculated from the resistance data. Prism RC-1 was dried to 105 C, and prism RC-2 to 60 C. Both specimens were tested through ten cycles of drying and wetting with water. Specimen RC-1 was soaked in a 1.0*N* sodium chloride solution after the tenth drying and was tested for one additional drying phase.

DISCUSSION

The data should be reviewed in the light of the stated objective of accuracy to 0.25 per cent moisture content, although, since the field condition usually covers the wetter region where shrinkage is not so great, it may be that the limit could be raised to 0.5 per cent or so.

The problem of correlating moisture content and shrinkage has not been treated here. Some early shrinkage measurements were made on some of the disks in order to decide on ranges of moisture content to be investigated. The results are similar to those of others, showing an irreversibility, especially on the first cycle, and a wetting-drying hysteresis, especially with the limestone aggregate. These factors of calibration and the change of the water content - shrinkage relationship with the age of the concrete and with its history must be kept in mind.

Certain trends are shown clearly by the data. The sensitivity of apparent capacitance to moisture changes is markedly affected by frequency. At low frequencies it is restricted to low water contents. Other factors remaining constant, the range of sensitivity increases with frequency. For the conditions of these tests, the sensitive range at 10^6 cps extended to at least 6 per cent for the first cycle and in most instances up to 8 or 9 per cent for subsequent cycles. When the disks were soaked in the salt solution, the sensitive range was restricted to water contents below about 6 per cent. At 60 cps the sensitive range was restricted to water contents of less than 2 per cent in all instances.

There was a shifting of the curves with subsequent cycles of drying and wetting, and there was a distinct hysteresis in the drying-wetting cycle. Both the hysteresis and shifting appear to become less pronounced upon subsequent cycles.

The hysteresis occurred to some degree in all instances, but the marked shifting of the curves on subsequent cycles occurred only with thin disks dried to 105 C. For disks dried to only 60 C and for the thicker disks regardless of their maximum drying temperature this tendency was greatly reduced.

A consequence of the dielectric properties of systems such as those studied here and of the experimental setup is that the calculated value of the capacitance of the specimen is subject to an increasingly large error when the conductance of the specimen becomes high or when the contact capacitance, that between the specimen and the electrodes, becomes large compared with that of the specimen.

Considering the 10^6 cps data, at the higher water contents these curves are controlled almost completely by the conductance of the disks and the contact capacitance between the disks and the electrodes. The dielectric constant of the disk has a negligible effect at the high water contents. The capacitance of disk CT-2 should be about two-thirds that of C-1 for the same water content, assuming dielectric constants to be approximately equal for equal water contents. However, the curves show that the apparent capacitance for disk CT-2 is the larger at the high water content. This can be the case only if the contact capacitance for CT-2 is the greater of the two and if the resistances of the specimens are low enough to make the apparent capacitance approach the contact capacitance.

In order to calculate the capacitance of the specimen proper, it is necessary to know the value of the contact capacitance. This value can be estimated with reasonable accuracy from the asymptote to the relative capacitance values at high water contents. When this is done, for example, for disks C-1 and CT-2 the resulting values of contact capacitance are 300 $\mu\mu$f and 320 $\mu\mu$f respectively.

The curve for C-1 after salt saturation is much higher than even that for the first drying cycle and gives additional evidence of the dependency of these values on the conductance of the specimen.

For the extreme conditions of drying a thin disk to 105 C and soaking in a 1.0N sodium chloride solution, the range of water contents at which the same apparent capacitance can be obtained is wide, being about 5 per cent at high water contents. However, for the less severe conditions of 60 C drying and for a thick disk, as represented by disk CT-2, the range is only about 0.5 per cent up to water contents of about 6 per cent and around 1 per cent at the highest water contents.

The conductivity, whether measured at 10 cycles or 10^6 cycles, shows about the same general trends of irreversibility and hysteresis as does the capacitance.

The same tendency of a lesser spread of values at the gentler drying condition is also present. The spread is somewhat greater for the 10 cps results than for the 10^6 cps results.

The relatively larger variations in the low-frequency dielectric conductivity are not so much the result of shifting of the data with successive cycles of drying and wetting as of a larger hysteresis in the cycle. Holmes (19), working with porous sandstone, investigated the influence of evaporation at the surface of the specimens during resistance measurements. He found that the distribution of moisture over the cross-section of the core is important, and whether or not evaporation is permitted has a pronounced effect on the results. This factor may contribute to the greater hysteresis found in the low-frequency measurements. Evaporation could also be a contributing factor to the hysteresis in the permittivity results.

The conductivity test results support the contention that the curve shifting and hysteresis shown by the apparent capacitance data are largely the results of changes in dielectric conductivity; however, the relative dielectric constant - water content relationships show that other factors must also be acting as these curves also exhibit the same trends.

For the dielectric constant data to yield a relatively accurate result for water content, it is desirable to work in a region of relatively low conductance. At higher water contents the only control on this factor is the geometric proportions of the test capacitor. Unfortunately the same changes that reduce the conductance also reduce the capacitance and with it the relative accuracy of its measurement. Therefore, any field use of this method poses an optimization problem in the design of the sensing elements.

One inviting compromise is to rely on the apparent capacitance as a moisture indicator. This idea is supported by the relatively low scatter of the data and the ease of measurement of apparent capacitance. However, this approach invites difficulties in calibration because of the influence of contact capacitance and electrode geometry on the apparent capacitance. An attempt to make such a calibration for a field installation would involve formidable problems of duplication of test conditions.

Indirect calibration techniques could, of course, be used to establish an apparent capacitance - water content relationship. The conductivity and relative dielectric constant - water content relationships could be established by methods similar to those used in this study, and the apparent capacitance - water content relationship calculated for the sensing capacitors to be used in the field. This procedure would require that the contact and geometric capacitances of the sensing capacitor be known. The geometric capacitance could be determined by measurements on liquids of known dielectric constant. The contact capacitance could be determined using salt solutions if it is assumed or could be established that the electrode to liquid contact capacitance would essentially duplicate the electrode to concrete contact capacitance for elements cast in concrete. If this could not be established, the contact capacitance could be estimated by monitoring the first drying after the element was cast in the concrete.

This study has shown the dielectric properties of concrete to be sensitive to increases in the salt concentration of the pore water, to drying to relatively high temperatures during the first few cycles, and to the thickness of the specimen tested. These factors must be taken into account for the successful calibration and field use of this method.

Considering only the variability of the results obtained, there is little to choose among the several properties investigated as moisture indicators. The apparent capacitance, dielectric conductivity, and relative dielectric constant all show significant variations under certain conditions, and none can be used as an indicator of water content in concrete under all conditions. It is also true, however, that the largest variations occur only under extreme conditions. Hence a realistic evaluation can be made only with respect to the conditions under which actual moisture determinations are likely to be made.

In many applications—a concrete pavement, for example—the section would be relatively thick, would be dried under atmospheric conditions, and, in many localities, would not have salts added to the pore fluid. These conditions might correspond to those of the thick specimens dried to 60 C and subject only to leaching. The results for these specimens show a minimum of variation and shifting with water content on successive cycles of wetting and drying. Also, for many applications the maximum moisture content of interest would not exceed 6 or 8 per cent. For example, it is unlikely that the moisture content of a pavement would normally exceed 8 per cent. Under such conditions the dielectric constant, dielectric conductivity, or apparent capacitance as determined from 10^6 cps permittivity measurements could be used reasonably successfully as moisture indicators. The accuracy of the determinations would probably be better than ±0.25 per cent for water contents less than about 6 per cent.

The indicated accuracy for the dielectric conductivity as determined from low-frequency resistance tests is about ±0.5 per cent for these same conditions. This is as good as that claimed for some other methods and has the advantage of experimental simplicity—for example, low frequency and bare electrodes—compared with the other methods explored in this work.

CONCLUSIONS

Based on the information obtained in this study the following conclusions seem reasonable:

1. The relationships between the dielectric properties of concrete and its water content are not constant. They are influenced by factors other than water content—notably electrical frequency at which the measurement is conducted, salt content of the pore fluid, and environment under which the concrete is aged. However, the conditions that lead to large variations in the relationships between dielectric properties and water content are not experienced in many field applications.
2. For moderate environmental conditions the dielectric properties of concrete, as determined from permittivity measurements, are potentially capable of indicating the water content with an accuracy of ±0.25 per cent for moisture contents up to about 6 per cent. However, for these accuracies to be attainable the instrumentation must be carefully designed for the specific requirements of the system.
3. Low-frequency resistance measurements possess a potential accuracy of about ±0.5 per cent moisture content, but there are indications that this accuracy could be improved. Because of the relative simplicity of the method it should be studied further.

Acknowledgment:

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EFFECTS OF CURING TEMPERATURE AND CREEP CHARACTERISTICS OF EXPANSIVE CONCRETES*

By A. Klein[1] and V. Bertero[2]

Engineers have long recognized the potentialities of utilizing forces induced through internal expansion in hardening concrete in overcoming some of the less desirable properties of concrete. Europeans, particularly in France and the USSR (**1–5**),[3] have conducted many investigations and made valuable contributions in the field of chemical reactions that produce internal expansion during hardening of concrete, as well as in the application of such "expansive cements" to special cases of construction and, in a limited degree, to the fabrication of structural elements.

In general, the early French investigators and more recently investigators in the USSR have used reactions developed during early hydration of a mixture of portland cement, calcium aluminate (*ciment fondu*) cements, and either gypsum or plaster of Paris. More recently, French engineers have utilized Lossier cement, consisting of a mixture of portland cement and the product of calcining a mixture of chalk, bauxite, and gypsum. In this case, the ground product of calcination, consisting of a mixture of calcium silicates, calcium aluminates, and hard-burned calcium sulfate (**3**), serves as the expansive component. Most recently, through a basic study of the chemical and solid-state reactions occurring in the high-temperature calcination of mixtures of oxides of calcium, aluminum, and sulfur, an expansive component was developed at the University of California and reported upon in 1958 (**6**). This compound was determined to be a calcium aluminosulfate clinker which, when ground and blended with portland cement, produced an expansive cement.

Continued research at the University of California resulted in the development of improved compositions which were found to be useful both in chemical prestressing or, with relatively small degrees of prestressing, in compensation of total drying shrinkage. In that research, considerable investigation was made of the expansion characteristics and compressive strengths of concretes containing expansive cement to evaluate the factors relating to control of the expansive reaction (**7**). From the results obtained in such investigation it was concluded that:

1. Either unrestrained or restrained expansions can be developed in concretes within ranges practical for structural concretes with the calcium sulfoaluminate expansive component used in this investigation. Restraint of expansion develops compressive stress in the concrete.

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper.

2. For a given curing condition, at any age the greater the unrestrained expansion the lower the compressive strength.

3. Even with the reductions in strength which accompany free expansion, it is possible with adequate restraint to produce self-stressing concrete of strength suitable for structural work. The greater the restraint the greater the strength.

4. For the concretes investigated, the higher the proportion of expansive component in the cementing material, the larger the ultimate expansion.

5. Magnitude of self-stress is affected not only by the amount of expansive component but also by the water-cement ratio of the mix, the curing conditions, and the percentage of steel employed in restraining the expansion.

Based upon the knowledge of expansive and restraining mechanics and of control variables gained from these investigations, further research was conducted, using selected structural elements including four chemically prestressed high-pressure pipes, a two-way-reinforced chemically prestressed slab, a corresponding one-way-reinforced slab, and a hyperbolic paraboloid thin shell (8,9).

The work on structural applications was conducted throughout at a temperature of about 70 F, both in manufacture and in curing. But from the standpoint of practical utilization of expansive cements, the effects of ambient curing temperatures—say between 45 and 100 F—upon the rate and magnitude of expansion and upon the physical properties of expansive concretes are of practical interest and significance.

Currently, structural precast elements are usually steam-cured under constant and predetermined conditions. On the other hand, in the case of concrete, mixed, placed, and cured in the field (airport and highway pavements for example), the temperature during curing may vary considerably. Curing temperature has a considerable effect on the properties of ordinary concrete, so it may be expected that it also plays an important role in the properties of expansive concretes.

In order to ensure that the great potentials of chemical prestressing will be achieved in practice, it is necessary to ensure adequate control of the factors that govern the expansive reactions, one of which certainly is curing temperature.

For prestressed concrete structural elements, in addition to the degree of prestress and the capacity to undergo stresses imposed by external loading, the potential loss of prestress through the effects of drying shrinkage and creep is a matter of great concern. Observations by the earlier foreign investigators, using expansive cements differing in chemical composition from those employed in these investigations, indicated highly unusual creep characteristics. In research on structural elements conducted at the University of California, curing was in fog or in water at 70 F for periods ranging between 20 and 35 days. Drying of slabs was at 70 F and 50 per cent relative humidity for periods between 30 and 60 days, and drying of the shell was in the laboratory atmosphere for 60 days. The magnitudes of stress losses in steel and in concrete due to drying shrinkage and creep were about equal to or less than those observed for conventional prestressed concretes. There is some evidence of a "pre-creep" mechanism occurring during the period of restrained expansion (7) which acts to reduce ultimate creep under external loading. However, it is considered desirable to evaluate creep characteristics of these expansive cements in order to predict stress losses reasonably for purposes of practical and economical design.

EFFECTS OF CURING TEMPERATURE

This phase of the investigation was designed to yield information on the effects of the temperature of water used in the curing of the expansive concretes. The study used 24 specimens, all of the same mix composition. Three groups of 8 specimens each were cast, each group being cured under water at a temperature of 45, 70, or 100 F, respectively.

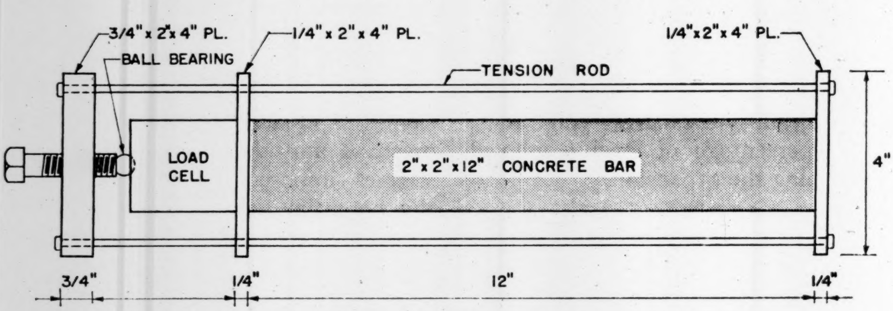

FIG. 1.—Uniaxially Restrained Concrete Bar and Frame.

Materials:

The cementing material consisted of 9.75 sacks of expansive cement per cubic yard of concrete. The expansive cement consisted of a blend of 75 per cent Type II portland cement and 25 per cent preground calcium aluminosulfate expansive component, by weight. A local sand (Eliot, fineness modulus 3.16) and a local gravel (Fair Oaks, fineness modulus 6.07) of maximum size ½ in. were used for aggregate. Forty-five per cent of the total aggregate was sand. The ratio of water to expansive cement was 0.30, and the ratio of expansive cement to aggregate was 1 to 3. A water-reducing retarder was used in the amount of 4 oz per sack of expansive cement. All concretes were mixed in a laboratory pan-type mixer, and mixing and casting of concrete for all specimens was at 70 F.

Fabrication of Specimens:

All specimens were 2 by 2 by 12-in. prismatic bars. Eight specimens were prepared for each curing temperature from a single batch of concrete. Of the eight specimens in each group, two were cured without restraint, and the remaining six were cured under uniaxial restraint.

The unrestrained specimens were cast in plastic molds and stripped 6 to 8 hr after casting. Stainless steel gage plugs were embedded in the end surfaces of specimens, to be used as reference points to measure the free expansion.

All specimens for curing under restraint were cast in steel molds. Stainless steel gage plugs were embedded in two opposite faces of each specimen at a gage length of 10 in. Soon after being stripped, the bars were inserted in the restraining frame, which consisted of two end plates and four tension rods (Fig. 1). The four tension rods were selected of a size to provide 1.1 per cent of steel reinforcement. The two end plates were brought into contact with the concrete bar by turning the adjusting screw; care was taken not to exert any significant pressure on the bar as indicated by the output from the load cell.

Curing of Specimens:

Immediately after being stripped, the specimens were subjected to water curing, the temperature of the water being kept constant at 45, 70, or 100 F. When it was observed that all specimens of the group had reached a state of tabilized expansion, the specimens were removed from the water and dried at 70 F and 50 per cent relative humidity.

Measurement of Expansion:

The rate and magnitude of the free expansion of the unrestrained specimens during curing were calculated from measurements by dial-gage extensometers at selected intervals of time. For

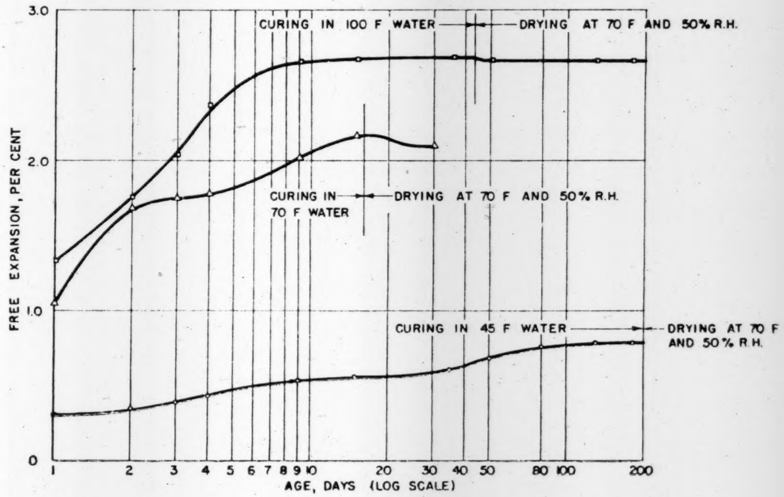

FIG. 2.—Effect of Curing Water Temperature on Free Expansion.

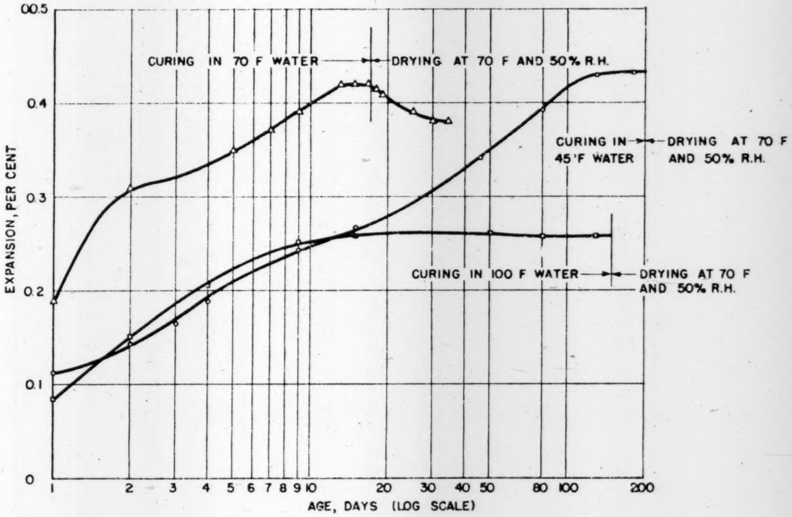

FIG. 3.—Effect of Curing Water Temperature on Restrained Expansion.

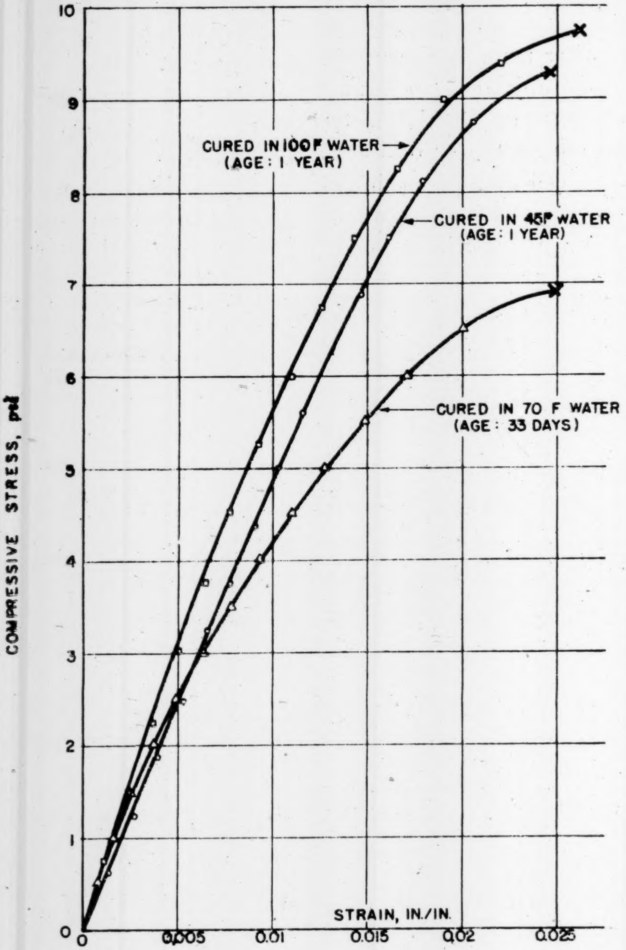

FIG. 4.—Effect of Curing-Water Temperature on the Stress-Strain Relationship of Restrained Expansive Concretes.

TABLE 1.—MECHANICAL PROPERTIES OF RESTRAINED EXPANSIVE CONCRETES.

	Curing at 45 F	Curing at 70 F	Curing at 100 F
Age at testing, days	365	34	365
Compressive strength, f_c', psi	9300	6940	9750
Secant modulus of elasticity, E_c, at $0.5 f_c'$, psi	4.8×10^6	4.7×10^6	5.8×10^6
E_c/f_c'	515	675	595
Poisson's ratio	0.19	...	0.20

the restrained specimens, Whittemore strain-gage data were obtained at the same intervals of time.

Calculation of the Amount of Prestressing:

The prestress developed due to expansion of concrete and restraint provided by the restraining frame was determined through the use of a special previously calibrated load cell (Fig. 1). The load cell was placed between the end plates and the adjusting screw of the restraining frame.

Expansion:

Expansions for unrestrained specimens as a function of time are shown in Fig. 2. Each curve is a plot of the average of the expansions for the two unrestrained specimens for each curing condition. The maximum stabilized unrestrained expansions were:

Temperature of Curing Water, deg F	Age, days	Expansion, per cent
45	200	0.78
70	16	2.17
100	12	2.72

Expansions for the restrained specimens as a function of time are shown in Fig. 3. Each curve is a plot of the average of expansions observed for the six specimens for each curing condition. The maximum stabilized expansions were:

Temperature of Curing Water, deg F	Age, days	Expansion, per cent
45	150	0.43
70	17	0.42
100	25	0.26

Mechanical Properties:

It is seen in Figs. 2 and 3 that expansion substantially ended and maintained a stabilized level with subsequent curing at the ages listed above. Specimens were removed from water at these ages and exposed to drying conditions at 70 F and 50 per cent relative humidity. After the contraction due to drying and shrinkage had reached a very low rate, each bar was sawed into three prisms, 2 by 2 by 4 in., which were then used to determine stress-strain relationships and compressive strength.

Unrestrained Specimens.—The compressive strengths of the small prisms cut from the specimens were:

Temperature of Curing Water, deg F	Age at Testing, days	Compressive Strength, psi
45	365	5600
70	33	1340
100	365	2100

Restrained Specimens.—The stress-strain relationships observed during the compression tests on the small prisms are presented in Fig. 4. Some mechanical properties are listed in Table 1 for the concretes for which stress-strain curves are shown in Fig. 4. Included in Table 1 for each of the three curing temperatures are the observed values of compressive strength and the calculated values of the secant modulus of elasticity, ratio of modulus of elasticity to compressive strength, and Poisson's ratio.

Magnitude of Prestress:

After a period of time under water, the load cells ceased to function properly, making a complete record unobtainable. However, as long as the load cells functioned properly, there was good agreement between values of restraining force calculated from observations with load cells and values calculated from observed expansions. For the given restraining process, after stabilization of expansion (including losses due to drying shrinkage), the final values of prestress are calculated to be:

Temperature of Water Curing, deg F	Prestress in Concrete, psi
45	1400
70	1260
100	850

Discussion of Results of Tests:

Unrestrained Specimens.—The data presented in Fig. 2 indicate that the higher the temperature of the water, the higher the initial rate and total amount of free expansion.

Also, as illustrated by the curves of Fig. 2, for specimens cured at 70 F and at 100 F, about 80 per cent of the total free expansion had occurred at 3 days. On the other hand, for specimens cured at 45 F, only 50 per cent of the total expansion was developed at 3 days. This difference indicates that relatively low temperature of curing water has considerable effect on the rate of reaction of the expansive component. The reaction is accelerated with higher curing temperature, a factor of primary importance in explanation of the expansion of restrained specimens.

Previous investigations (7) have shown that the compressive strength of unrestrained expansive concrete is closely related to the magnitude of expansion, decreasing as the total expansion increases. This relationship is confirmed by the results of the present study. If one considers the probable gain in strength that would have occurred in the specimens cured at 70 F and tested at 33 days, it may be expected that the compressive strength of the concrete of these specimens at an age of 1 year would have been slightly higher than that obtained for corresponding concrete cured at 100 F.

Restrained Specimens.—Since the total unrestrained expansion was higher for the concretes cured at higher temperature, it might be expected that the same expansion-curing temperature relationships would occur in the case of corresponding concretes cured with restraint. However, as noted in Fig. 3, such is not the case. Under restraint, maximum expansion was observed for concrete cured under water at 45 F, and minimum expansion was observed for concrete specimens cured at 100 F.

As pointed out earlier, results obtained with specimens of unrestrained concrete indicated that the major portion of the expansion when cured at 100 F took place at an early age, during a period when the concrete was weak and subject to plastic deformation. Hence, the concrete was not capable of exerting adequate pressure against the end plates of the restrained frame inasmuch as only uniaxial restraint was available. Consequently, the measured longitudinal expansion was small.

On the other hand, tests on unrestrained concrete cured at 45 F indicate that the expansive reaction took place at a rate considerably lower than that for the concrete cured at higher temperatures, and most of the expansive reactions occurred when the concrete had hardened sufficiently to produce considerable pressure against the end plates of the restraining frame, and without suffering significant plastic flow. These facts could explain the large amount of expansion observed in this case, compared with expansion of the concrete cured at 100 F.

As may be expected in view of this reasoning, the observed expansion for restrained concrete cured at 70 F (0.42 per cent) was greater than that for corresponding concrete cured at 100 F (0.26 per cent) and a little less than that for corresponding concrete cured at 45 F (0.43 per cent).

Compressive Strength of Restrained Concrete:

Although self-prestress developed by

the concrete cured at 45 F was consideradly higher than that developed by the concrete cured at 100 F, the compressive strength of the concrete cured at 100 F was a little higher than that of the concrete cured at 45 F. These relationships seem to confirm what already had been observed in previous investigations (7), that is, the mechanical properties of an expansive concrete that has been restrained during curing, with the restraint subsequently released, appear to be inversely related to the magnitude of expansion, within the range of restraint investigated. Considering the possible gain in strength with time that would have occurred in the concrete cured at 70 F and tested at 33 days, it may be expected that the compressive strength of this concrete at age 1 year would have been approximately the same as that for the concrete cured at 45 F.

For expansive concretes with potential for adequate prestress through restraint, it is reasonable to believe that there is some temperature that will be optimum with respect to the magnitude of self-stress and to compressive strength developed after stabilization of expansion. Such optimum temperature would be the temperature which, without reducing the magnitude of the expansive reaction, would delay the expansive reaction enough to permit the concrete to develop adequate strength and not be subject to excessive plastic deformation under conditions of uniaxial restraint. Based on the limited data available from these tests, this temperature may be between 45 and 70 F. However, more extensive studies should be conducted to determine not only this optimum temperature, but also a method to predict the amount of self-stress and compressive strength developed by expansive concrete cured at different temperatures.

Creep Characteristics of Expansive Concrete

As noted above, stress losses observed during drying shrinkage and creep of chemically prestressed structural elements (8,9) were at most equal and generally less than stress losses observed under corresponding curing conditions for conventional prestressed concretes stressed to about the same levels in steel and in concrete. Hence, although there was no investigation of creep during the curing and drying stages, it is believed that in view of the reasonably low stress losses, creep due to chemical prestressing is acceptably low. Actually there is some evidence that for externally restrained expansive concrete in 2 by 2 by 12-in. prisms there is a "pre-creep" mechanism during the first 4 to 6 days, while most of the expansion and self-stressing of steel and concrete takes place. It was observed (7) that specimens fog cured the first 7 days at 70 F, followed by 63 days' drying at 50 per cent relative humidity and 70 F, returned to their original lengths at age 7 days following drying shrinkage with 20 days of fog curing, again at 70 F. This may be considered as indicative of pre-creep, or plastic flow, during the period of high rate of expansion and low early-age strength.

The creep studies dealt with here concern only that due to external loading of 2 by 2 by 12-in. prisms self-stressed through the use of external restraint (Fig. 1). Both the portland cement and the expansive component were of the same stock of materials used in the investigation of the effects of temperature of curing upon the expansions of concretes. The compressive strengths of concretes at ages of external loading were determined, as before, by removing the uniaxial restraining equipment, sawing the 2 by 2 by 12-in. prisms into two 2 by 4-in. prisms (one from each side of the middle of length), and testing in compression.

Concrete Mixes:

Two concrete mixes were tested, both of high cement content suitable for chemical prestressing (Table 2).

Manufacture of Specimens:

Mixing was carried on first with dry ingredients, then wet, in a pan-type mixer. Concretes were cast in plastic forms, and the forms were then vibrated.

TABLE 2.—CONCRETE MIX DESIGNS FOR TESTS OF CREEP OF RESTRAINED EXPANSIVE CONCRETES UNDER EXTERNAL LOADING.

	Mix 1	Mix 2
Total cement, sacks per cubic yard	9	10
Expansive component, per cent of total cement	25	30
Water/cement ratio, by weight	0.31	0.31
Sand, per cent of[a] total aggregate, by weight	45	45
Water-reducing retarder, oz per sack	4	4
Maximum size of[b] aggregate, in.	½	½

[a] Fineness modulus Eliot sand, 3.16.

[b] Fineness modulus Fair Oaks pea gravel, 6.07.

The fresh concrete in the forms was then covered with a sheet of polyethylene film until the time of stripping.

The forms for the prisms of mix 1, providing four 2 by 2 by 12-in. prisms, were carefully stripped at age 7 hr. The prisms were then placed in the restraining rig (Fig. 1), and the wires were tightened until about 25 lb of total compression was acting upon the bars. The bars, with their restraining rigs, were then placed in fog at 70 F to allow curing and chemical prestressing under uniaxial force, for a restraining steel area of 0.88 per cent of the concrete cross-section.

Three bars were cast using concrete mix 2 and were stripped at 7 hr; the restraining rigs were mounted as in the case of mix 1, but the rigs were allowed to remain in the laboratory air until age 1 day, at which time bars under uniaxial restraint were placed in fog at 70 F, the restraining steel area in this case being 1.15 per cent of the concrete cross-section.

Loading of Specimens:

Mix 1.—The four bars were allowed to cure in fog at 70 F for 14 days, at which time expansion had been stabilized at 0.40 per cent. The bars were removed from fog and the restraint removed from one bar to allow cutting of prisms for compression testing. The average ultimate strength at age 14 days was 4650 psi. The remaining three bars were first relieved of restraint and then immediately reloaded in spring-loaded creep rigs and placed to dry at 70 F and 50 per cent relative humidity. Bars 1 and 3 were loaded with prestress plus 1150 and 1750 psi, respectively, while bar 2 was loaded only to the prestress level for use as a shrinkage "control." The amount of prestress force was determined from data provided by the load cells incorporated in each restraining rig.

Mix 2.—The three bars of this mix (bars 5, 6, and 7) also were cured 14 days in fog at 70 F and were then removed from fog and the restraining rigs removed. The stabilized expansion for this concrete mix was 0.57 per cent. Bar 6 was used for test of compressive strength, which was determined to be 3250 psi. (Fog-curing of these bars was begun at age 1 day.) In the manufacture of specimens with internal restraint and with adequate end anchorage, the problems related to stripping and time of start of curing are eliminated. For these tests the earlier the time of stripping and start of fog curing, the greater the prestress and the compressive strength of concrete subjected to external uniaxial restraint. Bars 5 and 7 were unloaded at age 14 days and then immediately reloaded in spring-loaded creep rigs and placed to dry at 70 F and 50 per cent relative humidity. Bar 5 was loaded only to the prestress level for use as the shrinkage control, and bar 7 was loaded with prestress plus 1200 psi.

All loaded specimens, including bars 1, 2, 3, 5, and 7, remained under constant load in the creep rigs for a period of 21 days. At the end of this period, bar 3 of mix 1 was unloaded to determine creep recovery, while bars 1, 2, 5, and 7 remained loaded for further study. A summary of conditions of curing, loading, and unloading is given in Table 3.

TABLE 3.—SUMMARY OF DATA COVERING CURING, LOADING, AND UNLOADING OF RESTRAINED EXPANSIVE CONCRETE CREEP SPECIMENS.

Mix	Bar	Age of Stripping and Application of 25-lb Load, hr	Age at Start of Curing, hr	Age at Creep Loading and Start of Drying, days	f_c' at Time of Creep Loading, psi	f_{creep} Sustained Creep Stress, psi	$\frac{f_{creep}}{f_c'}$	$f_c{}^a$, psi	$\frac{f_c'}{f_c{}^a}$	Age at Unloading Creep Specimen, days
1	No. 1	7	7	13.8		1150	0.25	1600	0.34	...
	No. 2	7	7	13.8		...	...	330	0.07	...
	No. 3	7	7	13.8		1750	0.37	2100	0.45	36.3
	No. 4	7	7	13.8	4650	...	...	...	...	...
2	No. 5	7	24	15.3		...	...	500	0.15	...
	No. 6	7	24	15.3		...	...	...	...	...
	No. 7	7	24	15.3	3250	1200	0.37	1780	0.55	...

[a] Total stress, prestress plus creep stress.

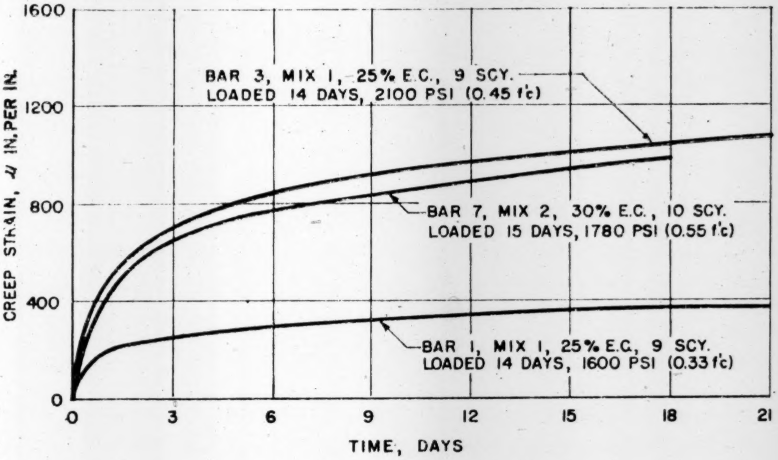

FIG. 5.—Creep Strain—Restrained Bars.

Strain Measurement:

Two methods for measurement of strains were used. Strains during expansion periods were measured with a Whittemore gage, while creep and shrinkage strains were measured with both a Whittemore gage and SR-4 wire strain gages. Gage points were located on opposite faces of the prisms and strain readings were averaged, theoretically to account for any bending induced by the spherical end blocks in the creep rigs.

Wire strain gages were bonded to the surfaces of prisms while curing in fog. Consequently, erratic readings occurred with some wire strain gages due to inadequate bonding in the presence of moisture or to moisture leakage to the gage in such a manner as to damage the gage. Where wire gages functioned properly, good correlation existed between strains by wire gage and by Whittemore gage on the same strain lines.

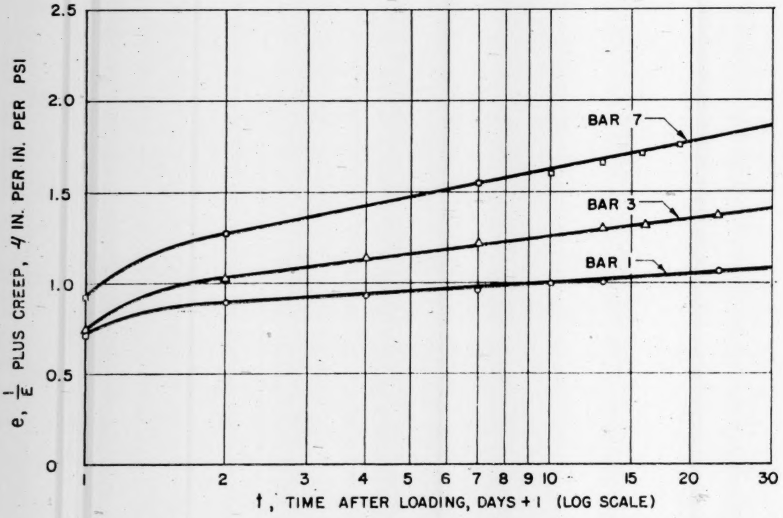

FIG. 6.—Total Axial Strains—Curves Fitting $e = \frac{1}{E} + F(K) \ln (t + 1)$.

Results of Creep Tests:

Specimens not subjected to creep loading were used for shrinkage correction. Creep strains were determined by subtracting shrinkage strain for control specimens from total strain at the same age. The creep strains for bars 1, 3, 7, after correction for shrinkage, are plotted in Fig. 5 as a function of loading time. For the same bars, the specific strain—total strain per unit stress—is plotted in Fig. 6 against the loading age plus one day $(t + 1)$.

As noted before, maximum expansions for mixes 1 and 2 were 0.40 and 0.57 per cent, respectively. The minimum expansions for the two mixes were 0.33 and 0.52 per cent, respectively; for practical purposes the average expansions were 0.36 and 0.55 per cent. Such expansions are in a range economically feasible for prestressing, giving tensile stresses in steel between 100,000 and 150,000 psi initially and between 85,000 and 125,000 psi after stress loss due to drying shrinkage and creep due to prestressing. Table 3 shows, by the differences between total stresses, f_c, and sustained creep stresses, f_{creep}, that precompression before drying shrinkage was 450 psi in bar 1 of mix 1, 500 psi in bar 5 of mix 2, and 580 psi in bar 7 of mix 2. Consequently the results of these creep tests are of considerable interest, while of limited scope, in that they are of potential use in the design of chemically prestressed structural elements.

TABLE 4.—CONCRETE MIX DESIGNS FOR TESTS OF CREEP OF UNRESTRAINED EXPANSIVE CONCRETES UNDER EXTERNAL LOADING.

	Mix 3	Mix 4
Total cement, sacks per cubic yard	9	9
Expansive component, per cent of total cement	15	20
Water/cement ratio, by weight	0.33	0.33
Sand, per cent of[a] total aggregate by weight	45	45
Water-reducing retarder, oz per sac	4	4
Maximum size of aggregate, in.	½	½

[a] Fineness modulus Eliot sand, 3.16.

[b] Fineness modulus Fair Oaks pea gravel, 6.07.

TABLE 5.—SUMMARY OF DATA COVERING CURING, LOADING AND UNLOADING OF UNRESTRAINED EXPANSIVE CONCRETE CREEP SPECIMENS.

Mix	Cylinder	Age of Stripping and Start of Curing, hr	Age at Start of Drying, days	Age at Creep Loading, days	f_c' at Time of Creep Loading, psi	f_{creep}, Sustained Creep Stress, psi	$\frac{f_{creep}}{f_c'}$	Age at Time of Unloading Creep Specimen, days
3	No. 8	8	22	...	...	...	...	...
	No. 9	8	22	...	...	...	...	...
	No. 10	24	22	28	9100	1980	0.22	50[a]
4	No. 17	8	22	...	...	...	...	...
	No. 18	8	22	...	...	...	...	...
	No. 19	24	22	28[b]	7160	2650	0.37	...

[a] Cylinder 10 unloaded at this point for further investigation.

[b] Cylinder 19 unloaded at this point for further investigation.

The curves in Fig. 6 approximate straight lines after age $(t + 1)$ equal to 2 days. Hence the specific strain per unit time can be fitted to the equation of form

$$\bar{e} = \frac{e_{\text{total}}}{p} = \frac{1}{E} + K \ln (t + 1)$$

where:

$\bar{e}$ = specific strain, or total strain per unit stress,

p = the total stress (prestress plus sustained creep stress),

E = the instantaneous elastic modulus, and

K = the creep coefficient, or slope of the straight line.

For the relationships plotted in Fig. 6, values of K are calculated to be:

For bar 1 of mix 1	0.18
For bar 3 of mix 1	0.32
For bar 7 of mix 2	0.53

Utilizing the values of K calculated for the three bars, it is estimated that at age of loading $(t + 1)$ equal to 1000 days, the ultimate creep strains, $k \ln (t + 1)$, would be:

For bar 1 of mix 1	0.54
For bar 3 of mix 1	0.96
For bar 7 of mix 2	1.59

Therefore, it may be stated that for the two bars of mix 1 the ultimate creep strains are somewhat less than the instantaneous elastic strain and that for the one bar of mix 2 the ultimate creep strain is somewhat less than twice the instantaneous elastic strain. The values for bars 1 and 3 are definitely low when compared to a conventional concrete, which might be expected to have ultimate creep strain about 2.5 times the instantaneous elastic strain. It is also noted that the value of ultimate creep strain for bar 7 is low, inasmuch as the total stress for this specimen was 55 per cent of the ultimate strength. The creep values noted would prove most useful in applications to chemically prestressed members in that the low creep would result in reduction in the stress loss.

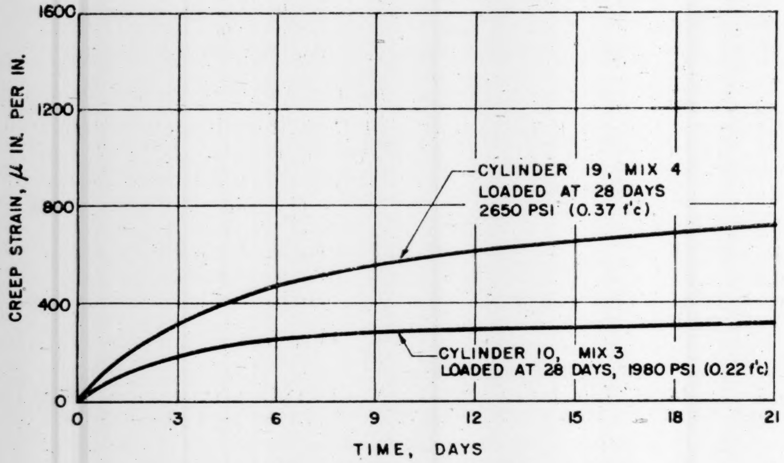

FIG. 7.—Creep Strain—Unrestrained Cylinders.

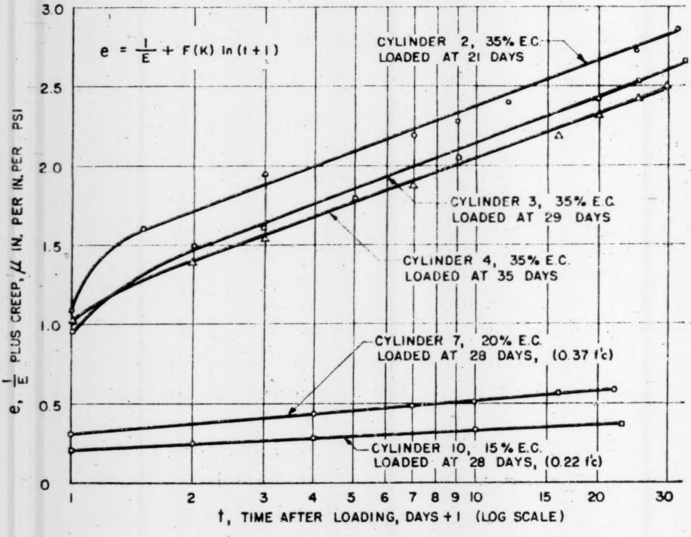

FIG. 8.—Specific Strain—Unrestrained Cylinders.

Bars 1 and 7 are still under load for further observation. Bar 3 of mix 1, unloaded at age 36 days, indicated a recovery of creep strain amounting to about 170 μin. per in. in 5 days. This bar was stored for further observation.

Creep of Unrestrained Concretes:

An additional investigation is under way for a study of unrestrained expansive concretes containing a smaller quantity of expansive component and having unrestrained expansions of 0.2 and 0.4 per cent, respectively. These mixes, with a total of 9 sacks of cementing material per cubic yard of concrete, were designed to be very strong and to have high elastic moduli. The creep specimens, 3 by 12-in. cylinders, are still under load for very late age observations (Table 4).

TABLE 6.—RELATIONSHIPS BETWEEN AMOUNT OF EXPANSIVE COMPONENT PER CUBIC YARD OF CONCRETE AND EXPANSION, STRENGTH, AND ULTIMATE CREEP.

Mix	Expansive Component, sacks per cubic yard	Maximum Expansion, per cent	Ultimate Strength, f_c', psi	Creep Coefficient, K	Estimated Ultimate Creep Strain, μin. per in.	Approx. Ratio of $e_{ultimate}$ to $e_{instantaneous}$	$\frac{f_{creep}}{f_c'}$
2	3.00	0.57	3250	0.53	1.59	2	0.55
1	2.25	0.40	4650	0.25	0.75	1	0.45
Mixes 3 and 4, average	1.58	0.30	8100	0.17	0.51	2	0.30
Zero (conventional concrete) .		. . .	. . .	. . .	. . .	2.5	0.30

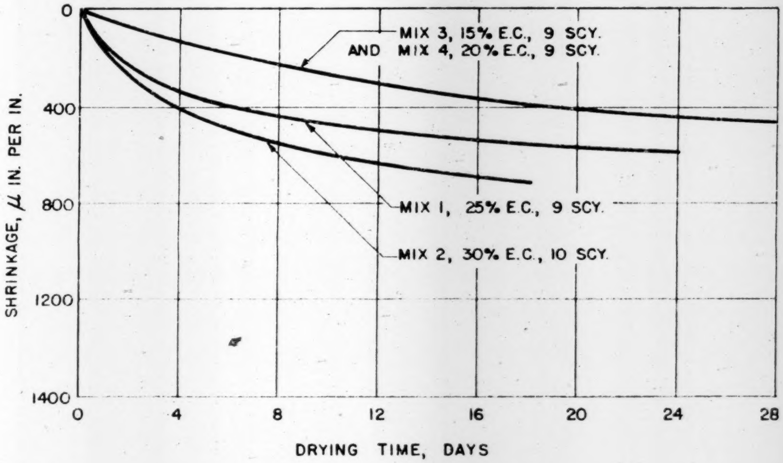

Fig. 9.—Shrinkage Curves for Mixes 1, 2, 3, and 4.

Table 5 gives the data pertaining to conditions of curing and creep loading for unrestrained expansive concrete cylinders. For each of concrete mixes 3 and 4 there were cast three cylinders 3 in. in diameter and 12 in. long, and for tests of compressive strength six cylinders 2 in. in diameter and 4 in. long.

The creep strain for cylinders 10 and 19, corrected for drying shrinkage, are plotted against days of loading time in Fig. 7, and the specific creep strains for these two cylinders are plotted in Fig. 8 against loading time plus 1 day ($t + 1$). Values of K, calculated from the data plotted in Fig. 8, are 0.13 for cylinder 10 of mix 3 and 0.21 for cylinder 19 of mix 4; and ultimate creep strains at 1000 days are estimated to be 0.39 and 0.63, respectively. Therefore, for these two mixes the average ratio of ultimate creep strain to instantaneous elastic strain is estimated to be equal to about 2.0. For practical purposes, it will be assumed that the average of the two mixes is a concrete containing 9 sacks of cementing material per cubic yard with 17.5 per cent of expansive component, or 1.8 sacks of expansive component per cubic yard of concrete.

Further, it will be assumed that the

average of values for mixes 3 and 4 will apply to ultimate strength, sustained stress, the ratio of sustained stress to ultimate stress, *K*, and ultimate creep strain. Table 6 lists, without regard to restraint or to size and shape of specimens, data pertinent to mixes 1, 2, and the average of values for the two mixes 3 and 4. With reference to the rate of expansion curves (Figs. 2 and 3), the expansive reactions which involve hydration of the expansive component are complete long before the application of external loads to the specimens. It is apparent that the higher the proportion of expansive component and the lower the proportion of portland cement, the more rapid is the hydration of the total expansive cement, all other things being equal. It is believed that if the ratio of sustained stress to ultimate stress had been 0.30 for all tests, the higher the content of expansive component in a concrete of given total content of cementing material, the lower would be the ultimate creep strains and the lower the

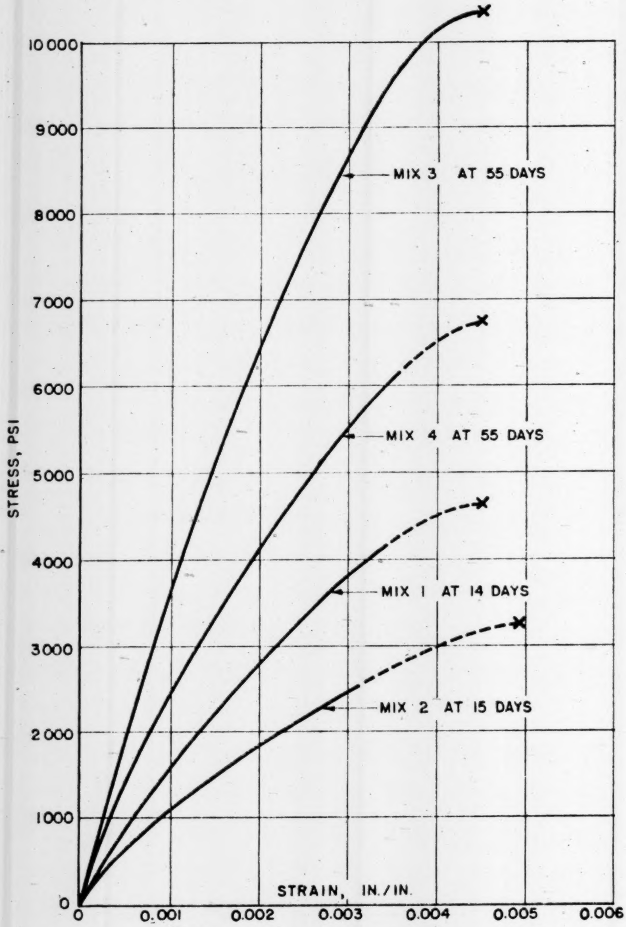

FIG. 10.—Stress-Strain Curves for Mixes 1, 2, 3, and 4.

ratios of ultimate creep strains to instantaneous elastic strains.

During the course of this preliminary study, it was noted that certain physical properties of these concretes did not appear to differ appreciably from what would be expected in a similar conventional concrete. Included among these properties are shrinkage, stress-strain characteristics, and creep recovery (Figs. 9 to 11).

The observed creep characteristics for all specimens tested and the data presented in Table 6 indicate that expansive concretes in the range of mixes studied and with maximum expansions below 0.6 per cent may be expected to exhibit ultimate creep strains due to external loads equal to or considerably less than those of conventional concretes subjected to the same ratio of sustained stress to ultimate stress at the same ages of loading.

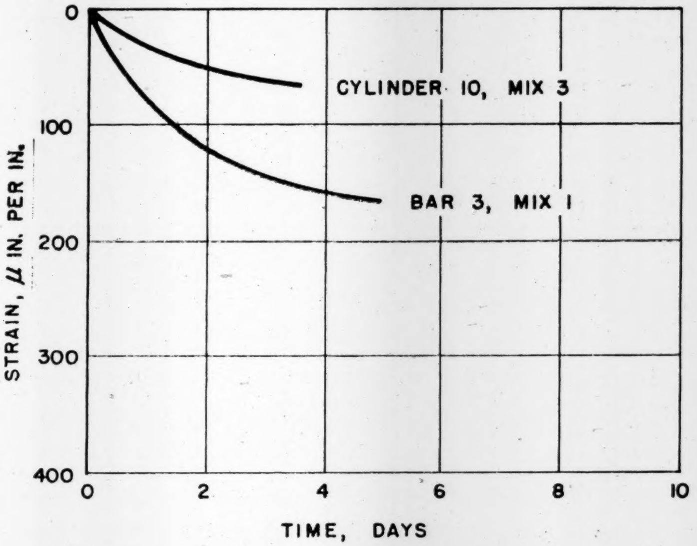

FIG. 11.—Creep Recovery

Acknowledgments:

Acknowledgment is made to the graduate students in the Division of Structural Engineering and Structural Mechanics, Department of Civil Engineering, University of California, Berkeley, for the research carried on by them under faculty supervision as part of their work toward the degree of Master of Science. The reports utilized in this paper include:

1. "Investigation of the Parameters Influencing Control of Expansive Reactions in Self-stressing Concrete," *SESM No. 5*, January, 1961, by George Chow.
2. "A Study of the Creep Characteristics, Under Compression, of Expanding-Cement Concrete," *SESM No. 20*, May, 1961, by Philip C. Warriner.
3. "Testing and Analysis of Self-stressed Concrete Elements," *SESM No. 23*, May, 1961, by Stephen H. Tso.

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Civils de France, Nos. 3–4, March–April, 1948, pp. 189–225.

(2) V. V. Mikhailov, "New Developments in Selfstressed Concrete," *Proceedings*, World Conference on Prestressed Concrete (San Francisco, Calif.), University of California, Berkeley, Calif., July 1957, pp. 25-1 to 25-12.

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(4) M. E. Perre, "Ciments sans retraint, ciments expansifs" (Nonshrinking Cements, Expansive Cements), *Cours*, Conference du Centre de Perfectionnement Technique, Vol. 2, April, 1944, pp. 26–34; *Cement and Lime Manufacture*, Vol. 24, July, 1947, pp. 75–77.

(5) F. E. Jones, "The Calcium Aluminate Complex Salts," Symposium on the Chemistry of Cements, Stockholm, Royal Swedish Institute for Engineering Research, 1938.

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(10) V. V. Mikhailov, "Stressing Cement and the Mechanism of Self-stressing Concrete Regulation," *Proceedings*, Fourth International Symposium on the Chemistry of Cement, Washington, D. C., 1960.

DISCUSSION

MR. W. R. LORMAN.[1]—Please clarify the distinction between the terms "creep" and "plastic flow." In the work of the Society's Committee C-9 on Concrete and Concrete Aggregates and American Concrete Inst. Committee 209, we have agreed that "plastic flow" is appropriate only when the stress (caused by the applied load) approaches the ultimate strength of the concrete.

MESSRS. ALEXANDER KLEIN AND V. BERTERO (*authors*).—The use of the terms "creep" and of "plastic flow" as used in this paper is in accordance with the definitions cited by Mr. Lorman. In the instances where the term plastic flow is used, justification of the term resides in the fact that at the early ages between casting and age 2 or 3 days the stresses created by restraint of high expansion are such that the stress-strength ratio is very high.

MR. WALTER H. PRICE.[2]—Have any tests been made to measure weathering resistance of expanding cements?

MESSRS. KLEIN AND BERTERO.—No tests have been made up to this time at the University of California with respect to freezing-and-thawing resistance of expansive concretes. Some very limited data are available for 3 by 4 by 16-in. stressed bars, manufactured by Concrete Research and Development Corp., for tests of freezing-and-thawing resistance using ASTM Method C 291[3] (rapid freezing in air and thawing in water).

The concrete was of nominal 7 sack expansive cement per cubic yard of concrete, the expansive component being 25 per cent by weight of expansive cement. The water:cementing material ratio was 0.32 by weight, maximum size of aggregate 1 in., and slump about 1 in. Concretes contained a water-reducing retarder and were placed by means of vibration. A control concrete was made without purposeful air entrainment, and an air-entrained concrete was manufactured with 5.2 per cent air content.

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[1] U. S. Naval Civil Engineering Laboratory, Port Hueneme, Calif.

[2] Riverside Cement Co., Los Angeles, Calif.

[3] Method of Test for Resistance of Concrete Specimens to Rapid Freezing in Air and Thawing in Water (C 291), 1961 Book of ASTM Standards, Part 4.

Restraint of expansion was provided by 0.57 per cent steel in the form of 3-wire high tensile twisted steel strand. Four strands were used per bar, one in each corner running longitudinally with $\frac{3}{4}$-in. concrete cover. No anchorages were used on steel strands, and since bond between concrete and steel provided the only restraint it was anticipated that there would be slippage at high steel stresses.

For the control concrete, without air entrainment, average expansion was 0.34 per cent and the average stress developed in the concrete was 270 psi. For the air-entrained concrete, the average expansion was 0.49 per cent and the average stress developed in the concrete was 170 psi.

In the control concrete without air entrainment, the loss in n^2 at 250 cycles of freezing and thawing was 18 per cent and the expansion 0.20 per cent. For the air-entrained self-stressed concrete, the average loss in n^2 at 960 cycles of freezing and thawing was 17 per cent and the average expansion 0.21 per cent.

Although these bars, for the purposes of test, were restrained longitudinally only and without anchorage, it is quite certain that such restraint will be neither adequate nor appropriate in applications to manufacture of structural elements.

REQUIREMENTS FOR FREEZING-AND-THAWING DURABILITY FOR CONCRETE

By W. A. Cordon[1] and D. Merrill[2]

Synopsis

Specification requirements for concrete durability have not changed commensurate with increased knowledge and research developed during the past 20 years.

This paper is a short review of current thinking on the causes of concrete deterioration due to freezing-and-thawing action. Laboratory test results made with a wide variety of materials and mix proportions are reported. A method for arriving at required cement contents and air contents based on required strength and required durability is discussed.

Recommendations for durable concrete have been consistent since freezing and thawing was recognized as a problem of concrete deterioration. The recommendations appearing in the 1940 Report of the Joint Committee on Concrete and Reinforced Concrete[3] (Table 1) limited the maximum water-cement ratios for concrete used in various exposure conditions and types of structures. This table has not been altered since even though the subsequent discovery and use of entrained air in concrete completely changed the concepts of what constitutes durable concrete.

Research into the basic mechanisms that cause concrete deterioration indicate that there are three principal phenomena that may cause concrete to deteriorate when exposed to freezing and thawing (**1–3**):[4] (1) buildup of hydraulic pressure in the gel structure of the cement paste, (2) growth of capillary ice during sustained cold periods, and (3) deterioration caused by concrete aggregates.

Accepted methods for producing durable concrete are simple and include the following:

1. Produce a high-quality portland-cement paste by limiting the amount of water in proportion to cement used in the concrete mix.
2. Use only high-quality aggregates that have satisfactory performance records in durable concrete.
3. Use the proper amount of air-entraining agent to produce an air-bubble spacing that will protect the concrete against the development of hydraulic pressure and growth of capillary ice.

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[3] Report of the Joint Committee on Standard Specifications for Concrete and Reinforced Concrete, *STP 5-A*, Am. Soc. Testing Mats., 1940, p. 12.

[4] The boldface numbers in parentheses refer to the list of references appended to this paper.

WATER-CEMENT RATIO

Portland-cement pastes of low water-cement ratio are more resistant to the buildup of hydraulic pressures and the growth of capillary ice simply because there are fewer capillaries in the paste. It has been estimated by Woods (4) that if a water-cement ratio below 0.42 by weight could be used in concrete, the small number of capillaries in the paste would not be vulnerable to freezing and thawing.

TABLE 1.—MAXIMUM PERMISSIBLE WATER-CEMENT RATIOS (GALLON PER SACK) FOR DIFFERENT TYPES OF STRUCTURES AND DEGREES OF EXPOSURE

Type of Structure	Exposure Conditions[a]					
	Severe Range of Temperature			Mild Temperature		
	In Air	At Waterline or within the Range of Fluctuating Water Level or Spray		In Air	At Waterline or within the Range of Fluctuating Water Level or Spray	
		In Fresh Water	In Sea Water or in Contact with Sulfates†		In Fresh Water	In Sea Water or in Contact with Sulfates
Thin sections, such as railings curbs, sills, ledges, ornamental or architectural concrete, reinforced piles, pipe, and all sections with less than 1-in. concrete cover over reinforcing	5.5	5.0	4.5[b]	6	5.5	4.5[b]
Moderate sections, such as retaining walls, abutments, piers, girders, beams	6.0	5.5	5.0[b]	...[c]	6.0	5.0[b]
Exterior portions of heavy (mass) sections	6.5	5.5	5.0[b]	...[c]	6.0	5.0[b]
Concrete deposited by tremie under water	...	5.0	5.0	...	5.0	5.0
Concrete slabs laid on the ground	6.0	...	...	...[c]	...	...
Concrete protected from the weather, interiors of buildings, concrete below ground	...[c]	...	...	...[c]	...	...
Concrete that will later be protected by enclosure or backfill but which may be exposed to freezing and thawing for several years before such protection is offered	6.0	...	...	...[c]	...	...

[a] Air-entrained concrete should be used under all conditions involving severe exposure and may be used under mild exposure conditions to improve workability of the mixture.

† Soil or ground water containing sulfate concentrations of more than 0.2 per cent.

[b] When sulfate-resisting cement is used, maximum water-cement ratio may be increased by 0.5 gal per bag.

[c] Water-cement ratio should be selected on basis of strength and workability requirements.

A water-cement ratio of 0.42 is generally not practical in concrete construction since a cement content of approximately 7 sacks per cubic yard would be required at usual water contents. Low water-cement ratios may be practical where very low slumps can be used or where water is reduced by chemical admixtures.

The theory that durable concrete can be produced by limiting the water-cement ratio is not confirmed by laboratory investigations. Powers (5) shows, for example, that even where there are fewer capillaries in the portland-cement paste, these capillaries must be protected by air voids. If this protection is not provided, the development of hydraulic pressures in the existing capillaries, although not as extensive as in paste of higher water-cement ratios, would be sufficient to cause deterioration. He also indicates that where paste is adequately protected by the proper amount of air entrainment, and where no capillary is more than 0.01 in. away from an air void, approximately the same durability exists with pastes of different water-cement ratios. Tests by Merrill (6) corroborate these results and show that in ordinary concrete poor

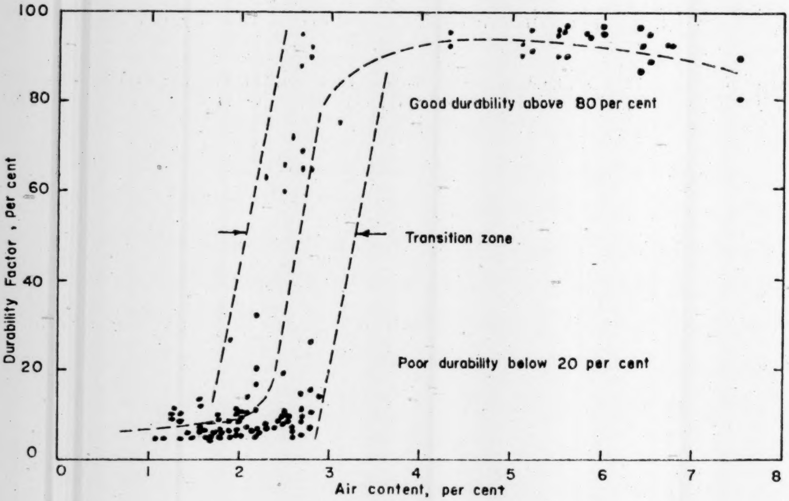

FIG. 1.—Durability of Concrete Containing Various Aggregates, Cement, and Air Contents, and Water-Cement Ratios.

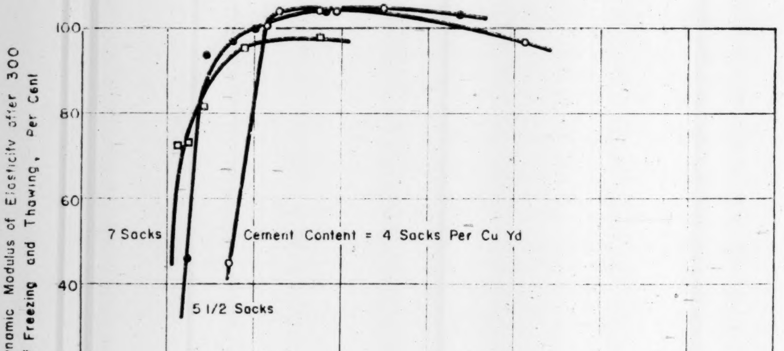

FIG. 2.—Durability of Concrete Containing 1½-in. Maximum Aggregate (After Klieger, 1956).

durability may be expected in any concrete containing less than 2½ per cent entrained air (Fig. 1). The points plotted in Fig. 1 are from concrete mixes with low to high water-cement ratios and containing four, five, and six sacks of cement. Tests by Klieger (7) show similar results and also indicate a transition zone of about 2 to 4 per cent air for durable and nondurable concrete even with differences in cement content from four to seven sacks per cubic yard (Fig. 2). It can be concluded that the water-cement ratio is not the principal criterion in producing durable concrete. But as Powers suggests, each capillary must have an air void sufficiently close to prevent the build-up of hydraulic pressures and to compete with capillary ice for gel water.

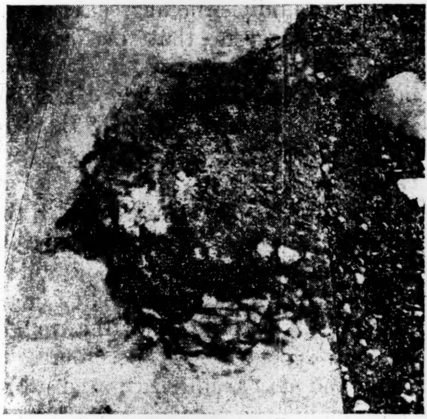

Fig. 3.—Deteriorated Concrete in Area Saturated with Dripping Water.

WATER-CEMENT RATIO VERSUS AIR ENTRAINMENT

The importance of air entrainment in producing durable concrete does not refute the theory that more durable concrete is produced with lower water-cement ratios. Tests by the U.S. Bureau of Reclamation (8) show that high-quality concrete (low water-cement ratio paste) produces concrete that is correspondingly more durable with air entrainment than low-quality concrete (high water-cement ratio paste) that is also protected with air entrainment.

Improvement of durability due to air entrainment is of far greater magnitude than the improved durability experienced when the water-cement ratio is decreased. For example the decrease in water-cement ratio from 6½ to 5 gal per sack may increase durability 100 per cent. The entrainment of approximately five per cent air in the same concrete, however, may increase the durability 1000 per cent.

WATER CONTENT

Additional water in excess of the amount required for hydration of the paste leaves voids within the hardened paste. The use of a minimum amount of water in fresh concrete is, therefore, desirable to reduce the number of these capillary pores. Low water content also increases the strength and decreases the shrinkage.

Shrinkage in concrete occurs as water is drawn from the gel pores of the cement paste, and is a function of the quantity of paste in the concrete. It is more desirable, therefore, to decrease the water content to produce a low water-cement ratio, rather than to increase the cement content. The water content of a concrete mix can be decreased by placing concrete at the lowest practical slump and by properly proportioning aggregates. Properly graded aggregates reduce the voids among the aggregate particles.

The water content of a concrete mix may also be reduced by the use of chemical admixtures commonly referred to as water-reducing admixtures. The admixture anions are absorbed by the cement particles, endowing them with negative electrical charges. Individual cement particles in the cement paste will repel rather than attract each other. During mechanical mixing of concrete, the cement flocs are broken up and the cement particles are dispersed, releasing about 10 per cent of the mixing water normally entrapped by the flocs. This permits a reduction of about 10 per cent of the average water content. Dispersing of cement flocs also densifies the cement paste and reduces the amount of uncombined water within the paste. This results in fewer capillary voids in the cement gel.

MOISTURE CONTENT IN HARDENED CONCRETE

One of the most important considerations in concrete durability is the moisture content or degree of saturation of the cement paste and aggregate particles. It has been established by many investigations that there is no freezing-and-thawing deterioration in concrete or aggregates that are not saturated. The expansion of freezing water in the capillaries of cement gel or aggregate voids containing less than 91 per cent saturation can be accommodated without developing harmful pressure. The portion of the slab shown in Fig. 3 that was not wet by dripping water, showed no signs of deterioration. The spot that was saturated during freezing weather disintegrated.

CONCRETE AGGREGATES

It is not possible to adjust the ingredients of a concrete mix to produce durable concrete if the aggregate particles expand, fracture, or force excessive amounts of freezable water into the paste as freezing progresses. The only apparent solution to the problem of nondurable aggregates is to limit the use of susceptible materials.

Sweet and Woods (9) found that certain aggregates caused deterioration of concrete regardless of the amount of cement or the construction practices used. They also suggested that in many cases standard aggregate tests are not adequate to detect such materials. The failure of standard tests to detect all materials that are not resistant to frost may be attributed to the following: (1) failure to consider the degree of saturation of the material at the time it is incorporated in the concrete, and (2) failure to duplicate the action of the mortar surrounding the aggregate particles. This suggests that performance tests for aggregates used in standard concrete mixes and exposed to freezing and thawing may be more realistic than tests of the properties of the aggregates themselves.

Lewis and Dolch (1) recognized the correlation between the absorption, degree of saturation, and durability and found that lack of durability of an aggregate in freezing and thawing is primarily dependent on its ability to become and stay highly saturated under given conditions of moisture. Recent specifications place a maximum limit of 3 per cent on aggregate absorption. This eliminates certain aggregates having poor durability in freezing and thawing although they may pass standard tests for sodium sulfate soundness and abrasion resistance.

In a study of chert and shale gravel in concrete, Schuster and McLaughlin (10) concluded that:

> Despite significant differences in the mineralogies, no difference was noted in the freeze-thaw durabilities of various chert samples. For all the cherts, significant deep-seated and surface deterioration occurred only in beams containing six to ten per cent of material with a bulk specific gravity (saturated surface-dry basis) of less than 2.45.
>
> Although the basic properties of the shales

varied even more widely than those of cherts, none of the shales caused deep-seated failure of the concrete. However, the most porous shales caused "popout" damage, which was especially severe at the six and ten per cent levels.

These conclusions indicate that, as far as the cherts used in the tests were concerned, the elimination of the lighter materials (specific gravity below 2.45) would produce a durable aggregate. In the case of shale only the most porous shale caused popouts. These results suggest the possibility of benefication of the chert sources and a method of rejecting shale aggregates by limiting the absorption to an acceptable maximum.

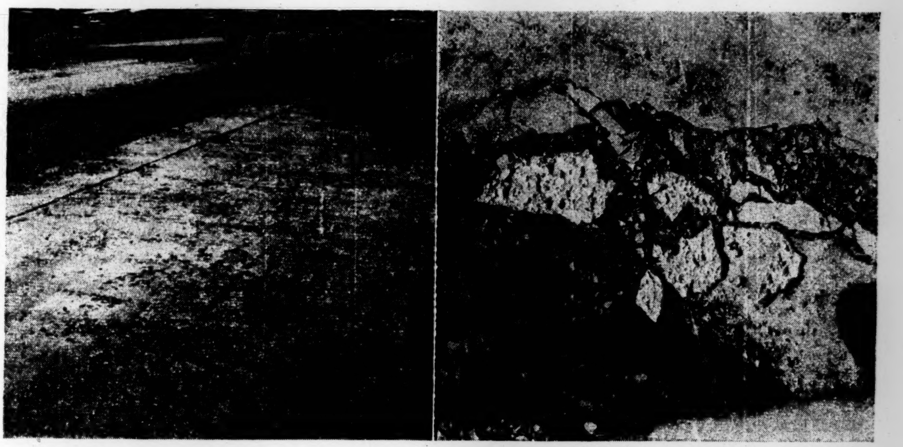

FIG. 4.—Scaling of Concrete Slabs During First Six Months of Service.

Heavy media separation is being used successfully by the U.S. Bureau of Reclamation in the construction of Glen Canyon Dam. The undesirable material in this case is a light chert of low specific gravity.

The experience in Michigan with aggregate benefication as reported by Leggs and McLaughlin (11) is typical of the problem facing many parts of the country as high-quality aggregate sources are becoming depleted.

Michigan natural gravel is now competing with imported high-quality coarse aggregates intended for concrete subject to severe winter exposure, as a result of installation of various types of benefication plants. Thus, local deposits are being utilized, after up-grading, without sacrifice of concrete quality.

. . . .

Benefication has enabled some gravel producers not only to survive competition, but to be able to furnish premium aggregates complying with rigid specifications for architectural concrete where severe exposure conditions are anticipated.

SCALING

Scaling of the surfaces of concrete slabs is one of the most insidious types of deterioration and may occur on slabs of highest quality concrete. There are two types of scaling: (1) salt scaling, which occurs on pavements where salt is used for snow and ice removal, and (2) the scaling that appears on concrete slabs after a particularly cold winter.

Salt scaling, as explained by Verbeck and Klieger (12), is caused by hydrostatic pressure of water in the voids immediately under the surface of the concrete slab. An increase in salt concentration in the capillary pores and subsequent freezing produces solutions of strong concentrations in the capillary voids. This tends to draw water to and increases the pressure in the capillaries. The additional pressure due to osmosis is believed to be sufficient to cause a tension failure of the concrete paste immediately under the surface of the slab. Salt also lowers the freezing point of water and increases the number of cycles of freezing and thawing during prolonged cold periods.

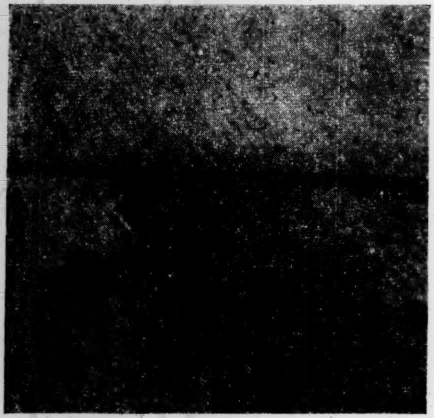

FIG. 5.—Lower Scaled Section of the Slab That Had Less Time to Dry Before Severe Cold Weather.

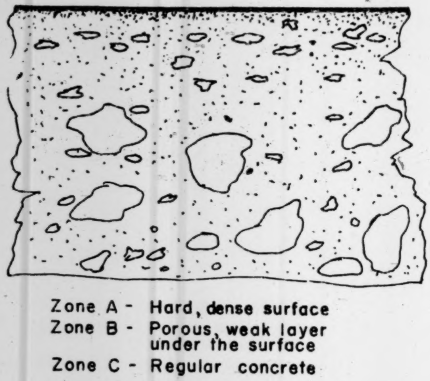
Zone A - Hard, dense surface
Zone B - Porous, weak layer under the surface
Zone C - Regular concrete

FIG. 6.—Overworking and Premature Finishing Creates Zones in Cross-Section Which May Be Responsible for Sealing.

The second type of scaling (Fig. 4) is not related to salt scaling. The concrete slabs shown were constructed late in the fall of 1961 and were made with a $5\frac{1}{2}$-sack mix placed at a slump of 3 to 4 in. Membrane curing compounds were placed on the surface of the slab as soon as it was finished to insure adequate curing. Approximately five months later, scaling occurred as indicated in the photographs. This type of scaling is typical of slabs exposed to severe cold periods before the concrete has had a chance to dry properly after curing. The concrete, placed late in the fall, was immediately sealed with a membrane curing compound and had little opportunity to dry (Fig. 5). The degree of saturation, consequently, was high during the subsequent prolonged cold period.

This discussion does not explain why the concrete slab did not merely expand as pressures developed rather than develop surface scaling. During finishing operations the surface of the concrete was densified and excess water was worked out of the paste. The capillaries in the surface paste were reduced. It is also possible that the concrete had not completely stopped bleeding when the surface was finished. Any small amount of additional bleeding water which developed after the finishing, accumulated under the surface "skin." This increased the voids immediately under the surface of the slab.

Three zones of different characteristics were produced with poor finishing practices (Fig. 6). Zone A had a dense, hard surface layer that resulted from excessive troweling, which brings fines to the surface and works out air voids and capillary voids. Immediately under the surface, zone B was deficient in cement and fines which have been worked to the surface by excessive finishing. This zone has an abundance of capillary voids, particularly if the slab was finished before all bleeding had stopped. This zone may also contain large air voids which usually bleed to the surface. Since a source of water to replenish gel water is nearby (at the surface), this presents ideal conditions for the growth of capillary ice in a saturated slab during freezing periods. Zone C was the regular concrete not affected by finishing.

One can conclude that finished concrete surfaces in a saturated condition are vulnerable to scaling. The ice in capillaries immediately under the surface skin grows to such proportions that the surface of the slab is forced off. An examination of Fig. 4 indicates that such a mechanism did actually force the surface from the concrete slab. Concrete immediately beneath the scaling was still in sound condition and apparently had not deteriorated. Scaling did not occur in concrete that was not exposed to surface moisture. This indicates that additional moisture from rain and snow was necessary to provide critical saturation.

The only difference between the concrete above the expansion joint, (Fig. 5), which did not scale and the concrete below the expansion joint, which scaled extensively, was the fact that the scaled concrete was placed approximately two weeks to one month later in the year. The concrete that did not scale may have dried out just enough to reduce the saturation to a point where the mechanism previously explained did not occur.

None of the concrete shown in Fig. 4 contained entrained air. One might logically conclude that if the proper amount of air had been entrained in the concrete, the slab would have been fully protected and scaling would not have occurred. There is evidence against such a dogmatic statement, however, since similar scaling has occurred in instances where the concrete contained entrained air. Apparently this type of scaling depends upon finishing methods and the drying time between placing and freezing.

PRODUCTION OF DURABLE CONCRETE

Extensive research by the concrete industry since the advent of air entrainment both in the areas of basic and applied research of field deterioration leads one to assume that a more realistic approach to production of durable concrete can be developed. Table 1 was developed by the Joint Committee on Concrete and Reinforced Concrete before air entrainment was known and is based upon control of the quality of paste. This does not necessarily produce durable concrete as previously discussed, and the entrainment of air is generally accepted as necessary to protect cement paste from freezing-and-thawing de-

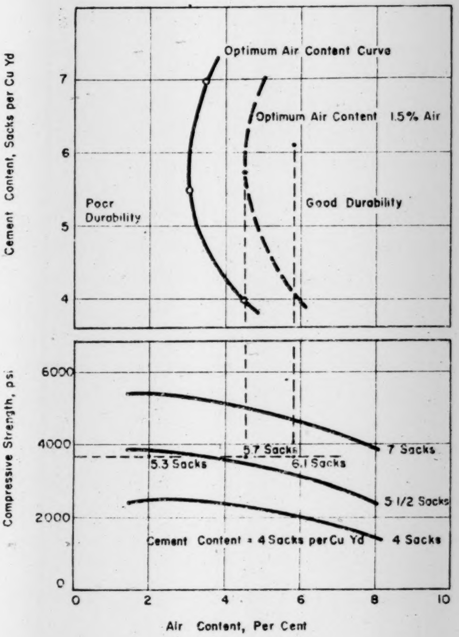

FIG. 7.—Relationship Between Cement and Air Content, Strength, and Durability.

terioration. There has been little effort, however, to correlate present-day knowledge to specific exposure conditions and available construction materials. Tremper and Spellman (13) made noteworthy progress in this regard by testing specimens of concrete made with job materials under saturation conditions and rates of freezing which had been measured in the field. It was found that a realistic appraisal could be made of aggregate sources which would be used in construction.

An attempt to correlate durability with air content, water-cement ratio, strength, and cement content is illustrated in Fig. 7.

A typical example will facilitate an explanation of the procedure of determining the cement content and air content required to produce concrete of adequate strength and durability. The upper curve is a plot of optimum air content as determined for $1\frac{1}{2}$-in. maximum aggregate by a series of freezing-and-thawing tests. The lower curves are determined by strength tests on cylinders of varying air content. Once these curves are obtained for given aggregates and cement, future concrete design is greatly simplified.

Assuming the required design strength of concrete is 3000 psi, based upon a quality control program it is known that an average strength of 3700 psi will meet the over design requirements for variations. Assume an air content 1.5 per cent in excess of the optimum air content to allow for mixing and placing variables. This will greatly reduce the possibility of the air content of the concrete falling into the transition zone (Fig. 1). The dashed curve in Fig. 7 represents air contents 1.5 per cent in excess of the optimum. Entering the figure at 3700 psi compressive strength, move right to a 5.3 sack mix (interpolate). Follow the 2 per cent air line vertically to the 5.3 sack mix in the upper part of Fig. 7. The indicated air content is below the optimum, and the concrete would not be durable. Repeating the process at a 6.1 sack mix and 5.8 per cent air, it is seen that the air content and cement content are in excess of the amounts required. A third trial at 5.7 sacks and 4.6 per cent air will provide the required strength and durability.

Standard methods for testing the resistance of concrete to freezing-and-thawing deterioration have received widespread criticism because they are often unrelated to field conditions. Powers (3) points out that rapid cycles of freezing and thawing build up hydraulic pressures out of proportion to slower freezing in the field, but, what is more important, do not allow for the growth of capillary ice which takes place during prolonged cold periods.

Freezing-and-thawing tests may serve several different purposes: (1) evaluate aggregates and cement, (2) evaluate admixtures, and (3) establish the relative durability of concrete.

Where the relative durability among various combinations of materials is all that is desired, testing procedures are not as important as when an attempt is made to establish the probable service of a particular concrete in a given location. In the latter case, correlation with field conditions is required.

CONSIDERATIONS FOR SPECIFICATIONS FOR DURABLE CONCRETE

In addition to the well-known requirements for the lowest practicable water content, slump, and water-cement ratio and air-entraining agents (14,15), the tests of this investigation indicate the following. More air entrainment is required in concrete mixes of low and high cement content than in mixes of medium cement content. It may not be practical to determine the optimum air content for each combination of materials but, as a general rule, tests indicate that where concrete is exposed to freezing and thawing in a saturated condition, it should contain a minimum of 5 per cent entrained air for concrete containing $1\frac{1}{2}$-in. maximum size aggregate (Fig. 7).

Special precautions for concrete slabs should include the following:

1. A minimum of finishing, which should be delayed until the concrete has reached its initial set and stopped bleeding. Avoid working a hard dense skin on the surface of exposed slabs.
2. A drying period, after curing, which will lower the degree of saturation to the lowest practical point before freezing weather.
3. Adequate drainage that will prevent moisture from standing on or around the slabs for extended periods, and
4. Elimination of inferior aggregate particles (16).

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DISCUSSION

Mr. Bailey Tremper[1] (*by letter*).—The purpose of this discussion is to cite instances of satisfactory performance of pavements constructed with aggregates having absorptions greater than 3 per cent in localities of severe freezing-and-thawing conditions, as shown by experience.

In the early 1930's, California constructed a substantial mileage of concrete pavement in the vicinity of Mt. Shasta. The road has since been reconstructed; however, substantial sections of the original pavement are still in service as local roads. The aggregates used were developed from a cinder cone and had absorptions of about 7 and 5 per cent in the coarse and fine aggregates, respectively. The pavement shows considerable wear in the wheel paths but otherwise is in good condition. There is no evidence of scaling or disintegration of the concrete. In contrast, a section of pavement a few miles to the north that was constructed with aggregates having absorptions of less than 3 per cent suffered severe disintegration. The difference in performance of the two sections appears to be connected with drainage or lack of it in the roadway sections. Reference is made to the second section merely to show that the exposure conditions are severe enough to cause disintegration of concrete due to freezing and thawing.

In 1958, California constructed about 7 miles of 4-lane pavement in the Sierras. The average absorption of the coarse aggregate was 2.4 per cent and that of the fine aggregate was 4.9 per cent. After 5 years of service there is no evidence of scaling or disintegration.

In 1959, California constructed about 10 miles of 4-lane pavement a few miles to the west and at a higher elevation than the 1958 pavement. The average absorption of the coarse aggregate was 3.0 per cent and that of the fine aggregate was 3.8 per cent. After 4 years of service there is no evidence of scaling or disintegration. Both of the latter two sections receive heavy applications of salts for ice control.[2]

The above illustrations indicate that poor performance in a severe climate does not necessarily result from the use of aggregates having absorptions greater than 3 per cent. Although an absorption greater than 3 per cent may be a danger signal, it appears unwise to reject aggregates merely because of high absorption.

Messrs. W. A. Cordon and D. Merrill (*authors' closure*).—The authors appreciate Mr. Tremper's comments regarding his experience with aggregates of high absorption. We agree that high absorption in aggregates may be only a danger signal. Porous aggregates with large pore spaces allow for the expulsion of freezing water. When the pores are small, hydraulic pressures build up within the aggregate particle, upon freezing, and cause failure. As Mr. Tremper points out, a specification which limits the absorption has the weakness of eliminating all aggregates of high absorption although some may be durable.

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[1] Consulting Engineer, Riverside, Calif.

[2] B. Tremper and D. L. Spellman, "Tests for Freeze-Thaw Durability of Concrete Aggregates," *Bulletin No. 305*, Highway Research Board, 1961, pp. 28–50.

A FIELD METHOD FOR SURFACE SOUNDNESS AND ADHESION TESTING OF CONCRETE*

BY C. V. WITTENWYLER[1]

SYNOPSIS

There is a need to evaluate the quality of new or old portland-cement concrete prior to application of surface overlays. It has recently become important to evaluate the soundness of thin surfacing overlays such as epoxy, polyester, latex cement, and bonded cement mortar on concrete roadways. No completely adequate test method has been available which will quickly and simply evaluate either the concrete quality or the overlay adhesion in the laboratory and in the field. A new concrete testing method is discussed which meets the need for a fast, inexpensive, convenient method to measure the tensile strength of concrete and the adhesion in tension of overlays to a concrete substrate. The method consists of bonding a two-in. diameter pipe cap to the surface under test. The pipe cap is then pulled in a direction perpendicular to the surface. The maximum force applied defines the breaking force, and the breaking force divided by the bonded area gives the strength in pounds per square inch.

NEED FOR A TEST PRIOR TO APPLICATION OF RESINOUS OVERLAYS

Epoxy resin overlays[2] are being used in increasing quantities by highway departments to provide a new, thin, lightweight, fast-curing surface for roadways and bridge decks. The overlay provides a highly skid-resistant, long-wearing surface which is resistant to deicing salts, freezing water, and to severe weather extremes.

Such epoxy overlays are made by spraying catalyzed liquid epoxy resins on a road surface (either bituminous or portland-cement concrete) at about $\frac{1}{4}$ gal per sq yd using methods and equipment similar to those used for conventional bituminous treatments. Then an excess of fine sand is added while the resin is still wet (Fig. 1).[3] When the resin hardens, the excess sand is swept off, leaving a sandpaper-like surface about $\frac{1}{8}$-in. thick. The hardening is achieved in 1 to 4 hr depending on the prevailing temperature. The completed overlay is highly resistant to gasoline, chemicals, and freezing weather (Fig. 1).

Since portland-cement concrete bridge decks represent a high initial cost, and since the maintenance burden for bridges is higher than for highways, bridges are now the largest end-use for epoxy resin surfacings. Bridges are also more susceptible to freezing-and-thawing damage because of the greater exposure for radiation and conduction heat losses and, as a result, undergo more freezing-and-thawing damage than highway surfaces. Salt is used more extensively to melt snow and ice on bridges than on most other highway surfaces, which frequently degrades the surface. A resinous overlay applied to bridge decks where the surface is unsound or degraded by deicing salts, freezing-and-thawing damage, or other causes, may fail when the weakened concrete fails in tension or shear. A knowledge of the actual physical properties of a surface to be coated is essential to avoid failure. A tensile surface soundness tester will quickly evaluate possible deterioration. It might be assumed that application to brand new concrete decks could eliminate this problem, but this has not been the case. Laitance is usually present, and its detrimental effect on adhesion makes the testing and pretreatment of new concrete just as important as the testing of old, exposed concrete.

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* Presented at the Fourth Pacific Area National Meeting, Los Angeles, Calif.

[1] Technical Service Laboratory, Shell Chemical Co., Union, N. J.

[2] C. V. Wittenwyler, "A Progress Report on Epoxy Road Surfacings," Highway Research Board Meeting, Jan. 10, 1962.

[3] U. S. Patent 3,033,088, May, 1962, Shell Oil Co.

The tensile surface soundness tester has also found use in epoxy resin overlays for industrial floors either to preevaluate the concrete or to test adhesion of the epoxy resin flooring.

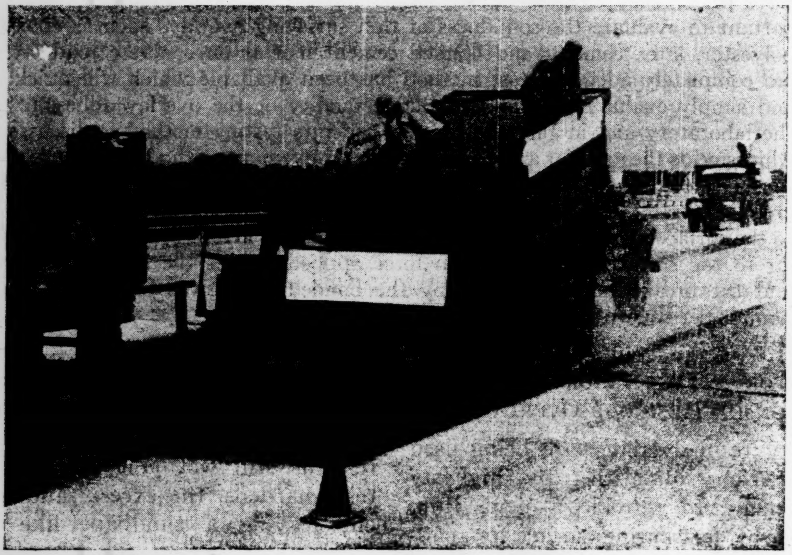

FIG. 1.—Application of Epoxy Road Surfacing to Bridge Deck.

DEVELOPMENT OF TENSILE SURFACE SOUNDNESS TESTER

This test method was developed six years ago, and since then has proved itself a valuable tool for field and laboratory evaluation of roadway surfaces and surfacing overlays. Some of the original resinous cement overlays for portland cement became loosened on exposure to traffic and weather and ultimately came off in large sheets. In such cases, the extent of the failure was initially determined by sounding (using a hammer) over the entire area. Although this sonic technique readily revealed areas that were loosened and blistered, it did not detect loosened areas that remained in close contact with the substrate. Such areas of secondary failure were readily detected later by using the improved tensile surface soundness test method.

A critical pre-examination of the surface condition is particularly important when a surfacing layer is to be bonded to the concrete surface, as well as to determine where sealing or resurfacing is impossible A weakened concrete surface could result in failure of an overlay through tensile failure of the bond. In addition, most concrete will have some "bleeding" or "water gain" at the surface, causing laitance. Such laitance will lead to weak concrete and poor bonding of a second layer of concrete to the material below. Even prior to painting concrete, a knowledge of the condition of the surface would be helpful to determine whether a surface pretreatment is required and the best type of paint for the prevailing surface condition.

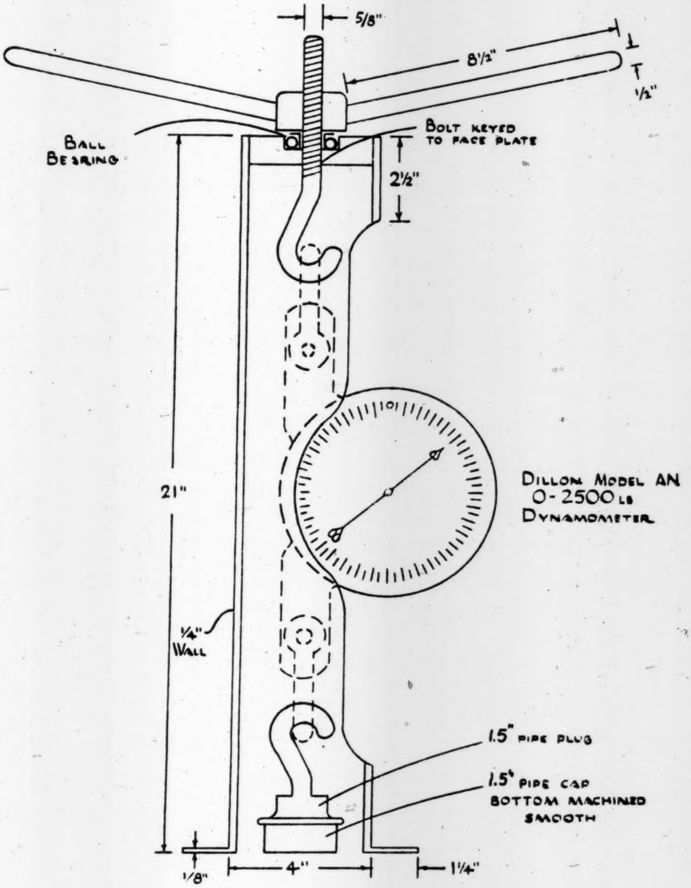
5/8"
8½"
½"
Ball Bearing
Bolt keyed to face plate
2½"
21"
Dillon Model AN
0-2500 lb
Dynamometer
¼" Wall
1.5" pipe plug
1.5" pipe cap
bottom machined smooth
1/8"
4"
1¼"

FIG. 2.—Tension Testing Unit with Dynamometer.

During the development period of epoxy road surfacing, it became quite clear that a successful overlay system would depend upon (1) an efficient cleaning method for concrete, (2) a high-speed application method to apply the overlay, and (3) a composition with good adhesion and integrity, both initially and after exposure to extremes of climate and wear. Many systems for cleaning and application were available, as well as variables in composition, but there were no adequate means of com-

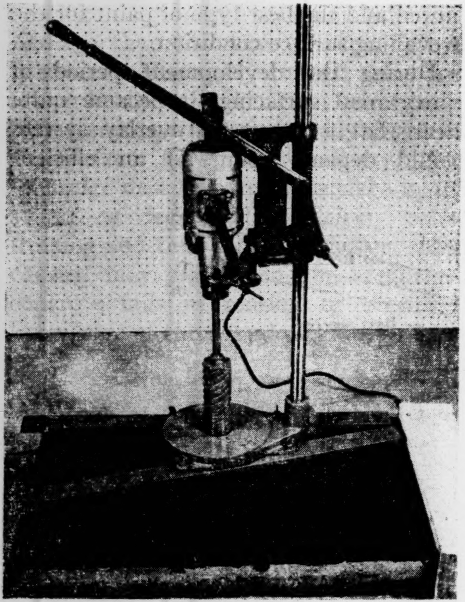

FIG. 3.—Core Drill Used to Cut Through Overlay Prior to Testing.

paring them or of measuring the effects of time, temperature, or exposure to wear on the stability of a given system.

To meet this need, the tensile surface soundness tester was developed (Figs. 2 to 7). The load is applied to a small area of the concrete. Surface effects such as laitance preclude measuring the full tensile strength of the concrete. In addition, as deeper failures occur, shear vectors develop. Hence, rather than tensile strength, a tensile index might be applied to the test results. A small island of epoxy overlay is isolated by cutting an annular hole through the surface to the concrete. A 2-in. internal diameter core bit is used for this purpose. A smooth-faced, 2-in. diameter pipe cap is then bonded to the surface with a quick-setting epoxy adhesive. The pipe cap is then pulled off the surface by screw action while the force applied is measured with a dynamometer (Fig. 2). The force to cause rupture and the type of failure which results is readily and quickly obtained with this method. Using this as a guide, surface pretreatment, application techniques, and composition variables may be compared. Periodic tests carried out in the field are used to determine the effect of weather and wear on the behavior of the resinous surfacing.

TENSION TESTING OF PORTLAND-CEMENT CONCRETE

Portland-cement concrete is in many ways an ideal material of construction. The cement is made from low cost, readily available raw materials—limestone and clay. Mixed with aggregate and water the resulting concrete sets rapidly and has excellent resistance to fire and has high compressive strength. However it is not uniform from lot to lot. Different batches of concrete in a given structure may show different strengths. Mix composition, temperature after placement, finishing techniques, curing conditions, exposure to wet freezing conditions, all have been known to lead to concrete with deficiencies in strength and expected performance.

As a means of inspection control, compressive strength tests are frequently run on concrete specimens. Although this is a meaningful test method, it does not always have the flexibility needed for field use. Compressive strength test cylinders may be cast at the time the batch is mixed, but this does not always reflect the curing conditions and cannot evaluate exposure conditions. Since compression specimens are usually 12 in. thick, a surface effect such as freezing-and-thawing exposure or laitance is not accurately revealed by the test.

To test the compressive strength of concrete after aging or exposure to traffic requires that a core specimen be cut from the concrete. To drill a 6-in. diameter core through concrete and to

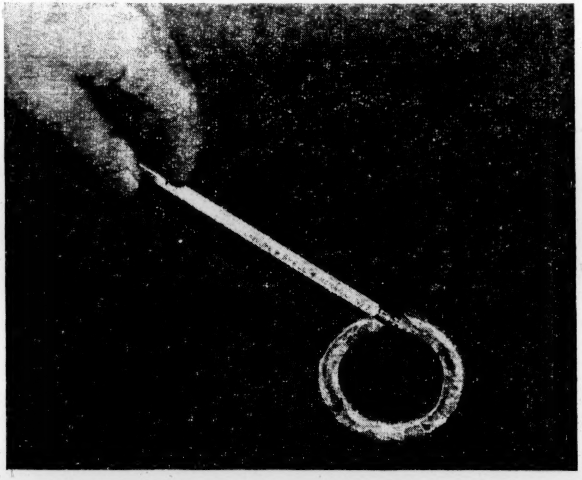

FIG. 4.—Isolated Test Area of Resinous Overlay.

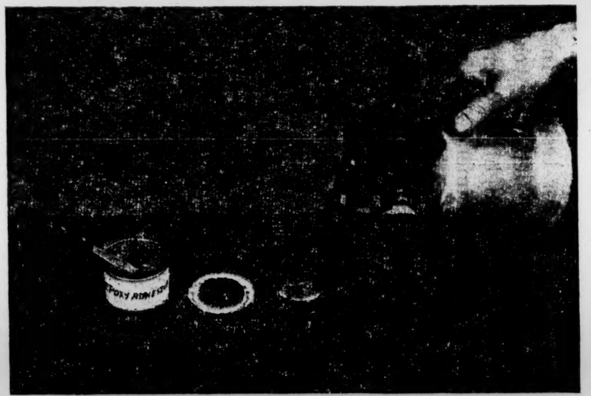

FIG. 5.—Bonding the Pipe Cap to the Test Area.

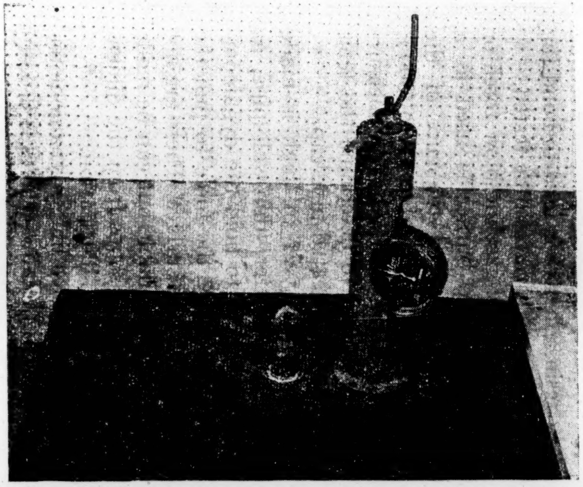

FIG. 6.—Tension Test Unit Used to Pull Pipe Cap from the Surface.

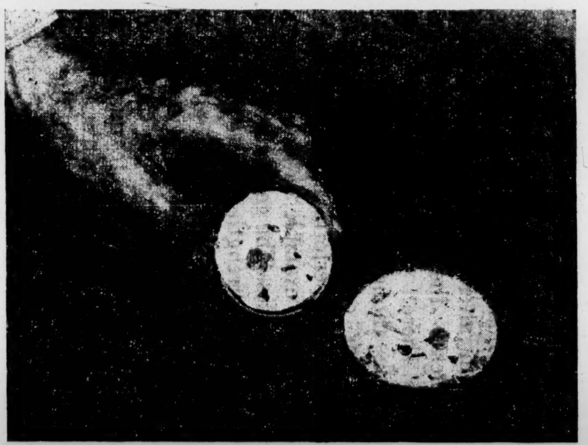

FIG. 7.—Pipe Cap After Pulling Showing Aggregate Split During Failure of the Concrete.

repatch the hole is a difficult and expensive undertaking. The inconvenience of such a practice precludes any testing at all in many cases.

As an alternative test of concrete soundness, the tension tester described here serves well. Tensile strength, though not normally determined for concrete, is an important factor in its performance. Cracking in concrete, for example, is a direct result of tensile forces. The general relationship of tensile strength to compressive strength of plain concrete is about 1 to 10.[4] Although this is not a fixed relationship, the tensile strength can readily be used to gage concrete quality. An advantage of this tension test is that it can easily be carried out in the field; unusual or critical conditions such as a weakened surface stratum can be determined in minutes. Additional testing may be continued to a final conclusion without a second field trip to the site to clear up conflicting results, as when core samples must be brought to the laboratory for testing. In addition, this method will detect a weak surface stratum which might be overlooked entirely if the specimen were capped and tested in compression.

TEST METHOD

The equipment is simple, inexpensive, and easily portable (25 lb) for use in the field as well as in a control laboratory.

Apparatus and Materials:

A pipe cap for 1½-in. nominal pipe is faced smooth on a lathe and a shoulder is cut to provide a 2-in. diameter surface. A hook 3½ in. long is made by welding a J-shaped piece of ½-in. diameter steel rod to a 1½-in. pipe plug which can then be screwed into the pipe cap.

A two-part, high-strength, fast-setting epoxy resin adhesive is used to bond the pipe cap to the surface to be tested.

A conventional gasoline blow torch or liquefied propane burner is used as a heat source to speed the cure of the adhesive, and a portable 0 to 500 F pyrometer fitted with a surface measuring head is used to determine the temperature of the heated pipe cap.

A jack housing 21 in. high is made from 4-in. diameter, thin-wall pipe, as shown in Fig. 2, to hold the dynamometer and to apply the force to pull the pipe cap from the surface. These are not commercially available but can be made by most machine shops from the dimensions shown in the illustration.

A maximum-reading dynamometer fitted with 1-in. diameter eyes is used to measure and record the force needed to pull the pipe cap from the surface.[5]

When the tester is used to measure the adhesion of an overlay to concrete, an island of overlay is isolated by using a core bit to cut through the overlay. This will eliminate any shearing action in the overlay. A tungsten carbide tipped 2¾-in. core bit is used to make the annular cut. The bit is driven by a 1-in. electric drill. A drill stand is used to steady the drill and insure more accurate and precise holes. A bench drill stand that operates on the drill press principle, as shown in Fig. 3, has been used successfully.

TEST PROCEDURE

Concrete Testing:

To test the surface soundness of concrete, a 4 by 4-in. area of the surface is cleaned to eliminate loose dirt by brushing with a wire brush. In the case of roadway surfaces, where oil is likely to be a contaminant, the surface is further cleaned by wiping with a clean cloth dipped in xylene or similar grease-cutting solvent. A prepared pipe cap is then bonded to the cleaned area. The torch is played over the surface of the pipe cap for about 10 to 15 sec to facilitate wetting and spreading of the adhesive. The adhesive is then spread on the cap and the cleaned concrete surface with a spatula. After positioning the pipe cap, the flame of the torch is directed inside of the pipe cap until the surface temperature reaches 275 F. This will require about 45 sec. By the time the pipe cap cools to ambient temperature (about 20 min), the pipe cap is ready to be pulled.

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[4] H. F. Gonnerman and E. C. Shuman, "Compression, Flexure, and Tension Tests of Plain Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 28, Part II, pp. 527–573, 1928.

[5] A Dillon Model AN, 0 to 2500 lb dynamometer, W. C. Dillon & Co., has been found satisfactory.

The hook is screwed into the plug and the jack housing placed over the hook. The dynamometer is connected between the upper screw hook and the lower plug hook. Force is applied by turning the screw at a rate so that the load as read by the dynamometer increases at about 100 lb every 5 sec. When the pipe cap separates from the surface, the maximum force recorded by the dynamometer is divided by the area in tension of the 2-in. diameter pipe cap (3.14 sq in.) to get the tensile strength in pounds per square inch.

After testing, the hole that results is patched by spreading a small amount of adhesive over the area and sprinkling excess sand over it. Cure can be speeded by playing the flame of the torch over the patched area for about 1 min.

Used pipe caps are recovered by burning the resin in a 1200 F muffle furnace. The sand or concrete residue is brushed off and the face lightly sandblasted to remove the layer of oxide formed by heating.

Overlay Testing:

In testing the adhesion of overlays to portland-cement concrete, the procedure is exactly the same except that a small section of the overlay is isolated by making a 2-in. diameter annular cut through the overlay to the substrate (Fig. 4). The cut is made just below the thickness of the overlay to avoid weakening the substrate. The dust and dirt are removed and the pipe cap bonded and pulled (Figs. 5, 6). Since a deeper hole is caused by this type of test than in concrete evaluation, it is more important that the hole be filled with sand and some of the epoxy adhesive (Fig. 7).

In using this method as a laboratory test, the jack housing and dynamometer may be replaced by a universal testing machine for convenience and greater accuracy.

LIMITATIONS

Six years of use of this tester have revealed some limitations. An original hydraulically operated model was replaced by the type described to provide a stepless, more uniform loading rate. Other limitations may someday be overcome by modifications in method or design. In the meantime, a knowledge of the limitations can prevent improper use of the results obtained with this method. Limitations for simple systems (evaluation of concrete) are not as significant as those for composite systems (evaluation of adhesion of overlays).

In simple systems the break occurs cleanly in only one stratum—the concrete. The results are interpreted by examining the tensile value obtained and the location of the break. If the break occurs at a low value, removing only a thin skin of cement from the surface of the concrete under test, it indicates that either a weak layer of laitance is present or, in some cases, surface damage from deicing salts or wet freezing-and-thawing cycling has occurred. Breaks extending $\frac{1}{8}$ to $\frac{1}{2}$ in. into the concrete at a high value signify sound concrete and concrete surfaces. Values up to 600 psi have been obtained with this type of a break, and quite frequently pieces of aggregate are split by the test, as shown in Fig. 7.

It is important to select the proper number of test sites as well as representative areas in the surface to be tested to obtain reliable results. For example, in testing roadways or bridge decks, at least one check should be made on each slab of concrete, and in evaluating surface soundness, tests should be made in curb areas, tire track areas, and in the zone between the tire tracks. Quite frequently, the concrete is sound in the tire track areas which are rapidly dried by traffic, but the accumulation of residual water and possibly salt lead to weakened surfaces and low tensile values in the curb area. Such weakened concrete must be removed by sandblasting, acid etching, or other means before applying an overlay to prevent premature failure.

In composite systems where overlays of epoxy, polyester, latex concrete, bonded mortar, oxychloride cement, or other materials are applied to concrete, failure can occur in three zones: (1) the concrete can fail in cohesion as in simple systems above, (2) the adhesive bond of overlay to concrete may fail, or (3) the overlay may fail in cohesion. Combinations of two or three types of failure sometimes occur. Adhesion failures over exposed aggregate are frequent and occur at a lower value than those over areas where aggregate is not exposed. These factors make the interpretation of results much more difficult than for simple systems.

The composite system being tested consists of a series of links in a chain, and only the weakest stratum is tested when the chain breaks. This has limited the use of the tension test when an attempt is made to compare the adhesive strength of a series of overlays. As overlay systems with increasing adhesive strengths are tested, a regular increase in tensile strength is shown, up to the breaking value of the concrete. Beyond that, no comparative results may be gained, since the concrete fails before the adhesive strength value is reached. This confines its use to below the breaking strength of concrete in tension, which may be as low as 250 psi.

Reports of test results with composite systems include breaking strength and a visual estimate of the percentage area of each type of break present; that is, cohesive concrete, cohesive overlay, and adhesive failures. Failures of composite systems in the overlay or in adhesion have limited meaning only if the failure occurs at a low value. Failure in the concrete at any level is normally expected and not objectionable. This ensures that the overlay is sound and has good adhesion. It may indicate low-strength concrete; however, the concrete strength is generally known before the overlay is applied.

In composite testing as in concrete testing, it is imperative that an adequate number of sites be tested and representative areas be chosen to ensure statistically reliable results.

CONCLUSIONS

With the growing use of resurfacings of many types for portland-cement concrete roadways and industrial floor surfaces, a need exists for a fast, inexpensive, simple, portable test method to measure the soundness of concrete and the adhesion of the overlay to the substrate. The method described here has been used successfully in over 100 applications in the field and in the laboratory to evaluate the quality of concrete, the adhesion of epoxy, polyester, and latex cement overlays, as well as adhesive for bonding new concrete to old. It has provided a means to determine quickly whether concrete is sound enough to justify treating with an overlay. It has also been used to compare the adhesion of different resinous overlays and to follow the strength changes in such systems after exposure to traffic, weather, and aging. With epoxy overlays of known strength, it has been used as a quality control method to determine whether the surface preparation and application techniques used meet the minimum specification requirements.

As a laboratory tool it has been used effectively to evaluate methods of cleaning concrete to remove oil, grease, laitance, and other foreign elements which would impair the adhesion of the overlay.

EFFECT OF SALT IN CONCRETE ON COMPRESSIVE STRENGTH, WATER VAPOR TRANSMISSION, AND CORROSION OF REINFORCING STEEL*

BY DONALD F. GRIFFIN[1] AND ROBERT L. HENRY[1]

SYNOPSIS

This paper presents data on the effect of salt in concrete on compressive strength, water vapor transmission, and corrosion of the reinforcing steel. Variables included (1) water-cement ratio, (2) diameter of specimen, (3) thickness of specimen, (4) per cent concentration of salts (either sodium chloride or sea-water salts), and (5) environment of specimen (20, 50, or 75 per cent relative humidity at 73.4 F).

The investigation, partially carried out using the wet cup method, has been in progress for approximately 2½ years. An additional study of experimental walls subjected to sea-water spray was begun about two years ago and will continue for several years.

Sea-water salts have been thought to be detrimental to reinforced concrete, but this study shows that, if controlled, some salt is beneficial. A gain in strength, lower values of water vapor transmission, and no significant corrosion of mild steel are realized when a salinity of 25 g of salt per kg of solution is established in the mixing water. Other results reveal that an electrical-resistance corrosion-rate detection probe appears to be a reliable device for measuring the degree of corrosiveness of any concrete environment to reinforcing steel; with or without salts, water vapor transmission varies directly with the water-cement ratio; thinner concrete specimens have somewhat higher transmission rates than thicker concrete specimens; with increasing salinities the effect of thickness upon water vapor transmission becomes more pronounced.

The purpose of the present investigation was to determine the effect of salt spray on portland-cement concrete and to determine the amount of salt permissible in the concrete mix. Specifically, it was desired to develop information about the effects of sodium chloride and of sea water on the strength and water vapor transmission of concrete, and the corrosive effect on the reinforcing steel. A previous investigation of water-vapor transmission of plain concrete using the wet-cup system has been described by the authors both in a Naval Civil Engineering Laboratory (NCEL) report (**1**)[2] and in the *Journal* of the American Concrete Inst. (**2**). Although more specific details about assembling wet cups with concrete specimens are given in references (**1**) and (**2**), the reader may obtain a good idea of

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[*] Presented at the Fourth Pacific Area National Meeting of the Society.

[1] U. S. Naval Civil Engineering Laboratory, Port Hueneme, Calif.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

TABLE 1.—SUMMARY OF CONCRETE MIX DESIGN DATA.

A. Characteristics of Materials

Cements: Colton, Type II; Colton, Type III; Victor, Type III.

Aggregates:
1. SG or San Gabriel, a reference aggregate from the Irwindale Pit of Consolidated Rock Products Co., Calif.
2. ENR or Eniwetok Atoll reef coral from Eniwetok Islet, Marshall Islands; a good-quality coral consisting of 95 per cent coralline limestone.
3. GMR or Guam reef coral from Apra Harbor, Guam, the Marianas; a poor-quality coral consisting of 96 per cent cellular coral or porous coralline rock.
4. Specific characteristics:

Item	San Gabriel		Eniwetok Atoll Reef Coral		Guam Reef Coral	
	C	F	C	F	C	F
Bulk specific gravity (oven-dry)	2.66[a]	2.63[a]	over-all average 2.49	2.49	weighted average L: 2.16 H: 2.11[b]	
24-hr absorption, per cent	1.6[a]	1.8[a]	2.32	2.32	20.0[a]	7.6[a]
Comparative general physical condition, per cent by weight	reference aggregate,		73 good	73	49 good	45
	approximately		25 fair	25	45 fair	45
	100 per cent good		2 poor	2	6 poor	10

[a] Determined by Task Engineers; data without [a] taken from *NCEL TN-335A*.

[b] L = water-cement ratio of 0.702, H = water-content ratio of 0.444.

NOTE.—San Gabriel aggregate was used unless otherwise indicated.

5. Grading: Pounds retained on each sieve per 6.0 ± cu ft batch:

Sieve	San Gabriel		Eniwetok Atoll Reef Coral		Guam Reef Coral	
	H	L	H	L	H	L
3/4	19.7	16.5	0	0	0	0
3/8	197.6	196.6	202.0	193.8	179.6	172.4
No. 4	125.1	121.0	116.4	110.0	103.4	98.0
No. 8	72.5	85.9	67.2	77.8	59.8	69.6
No. 16	65.9	82.9	61.2	75.2	54.4	67.0
No. 30	72.5	83.5	67.2	76.0	59.8	67.6
No. 50	59.2	76.2	55.2	69.4	49.0	61.8
No. 100	33.1	38.7	30.6	35.4	27.2	31.2
Pan	13.3	18.7	12.2	17.0	10.8	15.2
Total	658.9	720.0	612.0	654.6	544.0	582.8

Water: Port Hueneme tap water at 73.4 F.
Chemical analysis, ppm: hydroxide, 0.0; carbonate, 0.0; bicarbonate, 137.0; chlorides, 62.0; calcium, 38.0; magnesium, 14.6; sulfate, 465.0; sodium and potassium, 219.0.

Slump: Designed for 3 in.

B. Batch of 1.0 ± cu ft consisted of the following:

Mix Design Factors	San Gabriel		Eniwetok Atoll Reef Coral		Guam Reef Coral	
	H	L	H	L	H	L
Water-cement ratio	0.444	0.702	0.444	0.702	0.444	0.702
Water, lb[c]	11.7	11.7	13.4	13.8	13.4	13.8
Cement, lb	26.4	16.7	30.1	19.7	30.1	19.7
Cement factor[d]	7.62	4.81	8.65	5.66	8.65	5.66
Aggregate, lb	109.9	120.0	102.0	109.0	90.7	97.2

[c] The quantity of water added at the mixer was corrected for moisture present in the aggregate and moisture required for absorption.

[d] Sacks per cu yd of concrete.

the wet-cup system by referring to Figs. 10 and 13. In all cups, the water in the liquid phase of the cup was distilled water. All saline waters referred to are the mixing waters for the concrete. The vapor phase portion of each cup was initially flushed out with ambient air.

TABLE 2.—COMPRESSIVE STRENGTH VERSUS SODIUM CHLORIDE CONTENT.

NOTE.—Colton Type III cement; slump: 3 in. with no salt; with salt, slump ranged from 5 to 8 in. as salt content was increased.

Batch	Sodium Chloride[a]	Compressive Strength After Various Days of Cure in 100 Per Cent Relative Humidity at 73.4 F, psi[b]						
		14 Days	Standard Deviation, 14 Days	28 Days	56 Days	112 Days	224 Days	364 Days
WATER-CEMENT RATIO = 0.702								
S1L-1	0	3990	95	4480	4700	5030	4970	4910
S1L-2	0.075	4290	100	4780	5020	5170	5150	5060
S1L-3	0.150	4410	95	4890	5070	5290	5350	5230
S1L-4	0.225	4550	115	4790	5210	5460	5510	5420
S1L-5	0.300	4630	115	5020	5330	5630	5500	5450
S1L-6	0.450	4430	95	4800	4920	5160	5380	5280
S1L-7	0.600	4250	95	4610	4740	5080	5060	5330
S1L-8	0.750	4150	100	4510	4660	5010	5130	5150
S1L-9	0.900	4090	105	4440	4750	4930	4860	4850
S1L-10	1.200	3830	100	4200	4360	4540	4710	4630
S1L-11	1.800	3280	85	3620	3800	4040	4130	4450
S1L-12	2.400	2760	65	3000	3320	3750	3660	3960
WATER-CEMENT RATIO = 0.444								
S1H-1	0	6760	200	6970	8020	8060	8150	8450
S1H-2	0.075	6550	185	6930	7290	7880	8040	8440
S1H-3	0.150	6620	180	7380	7630	7940	8180	8420
S1H-4	0.225	6840	180	6950	7820	7800	8570	8630
S1H-5	0.300	6940	125	7320	7760	7650	8380	8570
S1H-6	0.450	6780	185	7210	7590	8010	8480	8800
S1H-7	0.600	6640	150	7180	7550	7910	8100	8160
S1H-8	0.750	6510	130	7260	7560	7370	7990	8410
S1H-9	0.900	6150	185	6460	6820	7740	8000	8000
S1H-10	1.200	5800	160	6330	6720	7000	7310	7630
S1H-11	1.800	5070	125	5390	5800	6240	6370	6260
S1H-12	2.400	4480	65	4770	5020	5340	5450	5150

[a] Per cent by weight of fresh concrete.

[b] Three cylinders (4 in. in diameter by 8 in. long) for each age for each salt concentration except for 14-day age: 15 cylinders for each were used.

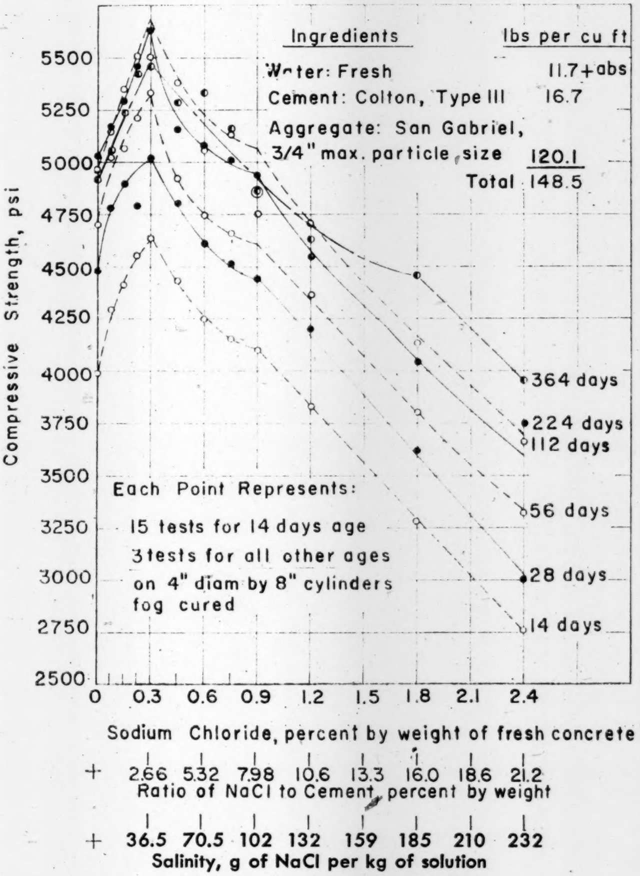
The previous study employed concrete specimen disks of one diameter and one thickness. In contrast, the study reported here includes disks of two diameters and of several thicknesses and involves a specimen series with varying concentrations of sodium chloride and a specimen series with varying concentrations of sea water in the mixing water.

A collateral study of small, experimental reinforced-concrete walls to measure their durability to marine exposure, particularly as related to corrosion of the reinforcing steel, was initiated in May, 1961, and will extend for several years. A description of this phase of the investigation and the observations to date are included in this paper as a related study.

CONCRETE MIX PROPORTIONS

The concrete mixes were proportioned according to the mix design data of Table 1. San Gabriel aggregate (described in Table 1) was used except where other aggregates are specified. Except for the reinforced-concrete walls, all batches had a volume of 2.25 cu ft and were mixed in a pan-type mixer, with the following sequential operations:

1. Add gravel, sand, and cement.
2. Mix dry for 30 sec.
3. Add water (73 F) in the shortest time possible with the mixer running.
4. Mix for 150 sec, for a total mixing time (items 2 to 4) of 3 min.
5. Take slump test (designed for 3 in.); if slump is too low, add water, mix for 30 sec, and repeat slump test.



(*a*) Water-cement ratio = 0.702.

FIG. 1.—Compressive Strength Versus Sodium Chloride Content.

(Continued on pp. 1050 and 1051.)

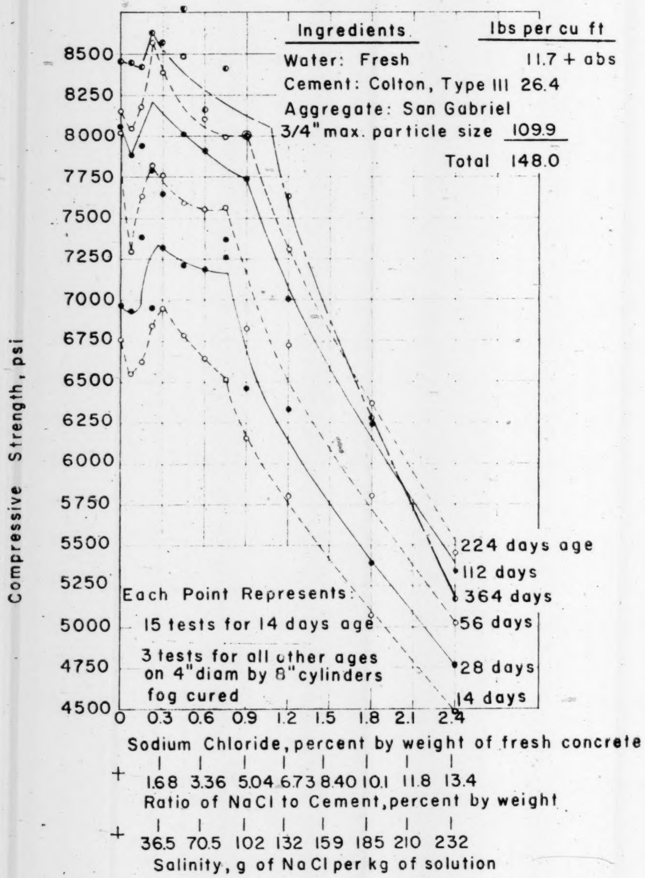
6. Place concrete in molds in ASTM standard manner, except vibrate with stud vibrator.

7. Smooth off tops with wooden float.

8 Cover with metal plates and place in fog room.

9. After 24 hr, strip molds, number cylinders, and place in fog room.

Concrete for the walls was mixed in a revolving-drum mixer using a batch of approximately 6 cu ft. The mixing procedure was similar to that used above except that the coral aggregate was saturated-surface-dry when placed in the mixer.



(*b*) Water-cement ratio = 0.444.

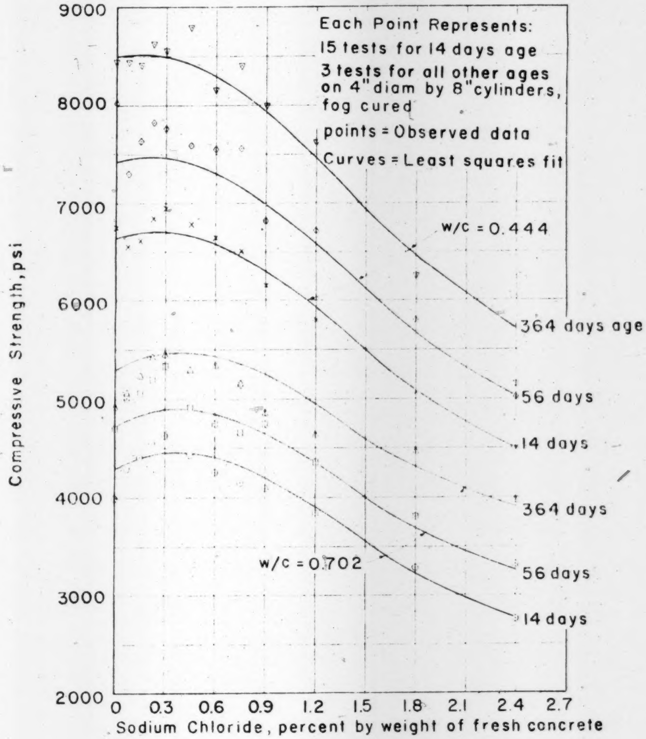
FIG. 1.—*Continued.*

Every batch and specimen was treated identically. The fresh concrete was vibrated with a 1-in. stud vibrator in three lifts, instead of rodding. Each lift was vibrated approximately 2.5 sec.

COMPRESSIVE STRENGTH

Sodium Chloride Series:

Previous attempts to determine the compressive strength of concrete as a function of sodium chloride content made use of data for batches of concrete made at widely varying times for different purposes (1). Series of data such as those shown in Table 2 can be satisfactorily obtained only by having an adequate supply of uniform materials and by batching in an orderly manner on successive days using the same personnel for each operation.



(*c*) Least squares fit.

FIG. 1.— *Continued.*

Figures 1(*a*) and (*b*) show graphically, for two different water-cement ratios, how compressive strength varies with increases in the sodium chloride content of the mixing water.[3] There is a pronounced and unmistakable optimum sodium chloride content for maximum compressive strength for each of the water-cement ratios. The optimum sodium chloride content for the two water-cement ratios is not a function of the ratio of sodium chloride to cement, as might be expected, but is more nearly a function of salinity (grams of salt per kilogram of solution) of the mixing water. The optimum salinity for maximum strength seems to be within the range of 30 to 36 g per kg for the two water-cement ratios.

Mixing water of the least salinity used (about 9 g per kg) increased the slump from a design of 3 in. to a value of 5 in. With increasing salinities, the slump increased to 8 in. A constant slump of 3 in. undoubtedly would have required less mixing water, with consequently

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[3] Sodium chloride, U.S.P. granular; F.W. (formula weight) = 58.45, dissolved in the mixing water.

TABLE 3.—CHEMICAL COMPOSITION OF HYPOTHETICAL SEA WATER.

Composition of Sea Water (3)		Combinations of Ions in Sea Water (4)	
Ion	g per kg	Salt	g per kg
Cl^-	18.980	NaCl	23.477
SO_4^{--}	2.649	$MgCl_2$	4.981
HCO_3^-	0.140	Na_2SO_4	3.917
Br^-	0.065	$CaCl_2$	1.102
F^-	0.001	KCl	0.664
H_3BO_3	0.026	$NaHCO_3$	0.192
Na^+	10.556	KBr	0.096
K^+	0.380	H_3BO_3	0.026
Mg^{++}	1.272	$SrCl_2$	0.024
Ca^{++}	0.400	NaF	0.003
Sr^{++}	0.0085	Total	34.482

NOTE.—The salinity of this hypothetical sea water is 34.325 g per kg as shown in reference (**3**).

TABLE 4.—COMPRESSIVE STRENGTH VERSUS SEA-WATER CONCENTRATION FOR WATER-CEMENT = 0.702.

NOTE.—Colton Type II cement; slumps: 3 in. and nearly constant for all batches.

Batch	Sea-Water Concentration[a]	Compressive Strength After Various Days of Cure in 100 per cent Relative Humidity at 73.4 F, psi[b]								
		7 Days	14 Days	28 Days	Standard Deviation, 28 Days	56 Days	112 Days	224 Days	364 Days	546 Days
S3L-1R	0	2130	2800	3560	150	4240	4820	5080	5160	5250
S3L-2	20	2290	2830	3330	45	4080	4770	5030	5370	5350
S3L-3	40	2390	2930	3460	45	4370	4690	5220	5290	5600
S3L-4	60	2590	3010	3610	45	4150	4700	5060	5280	5410
S3L-5	80	2670	3210	3770	70	4280	4810	5180	5460	5440
S3L-6	100	2800	3400	3890	100	4620	5080	5440	5760	5840
S3L-7	120	2820	3470	3980	65	4410	4950	5330	5350	5560
S3L-8	140	2840	3420	3810	110	4270	4780	5200	5340	5440
S3L-9	160	2830	3410	3820	100	4350	4860	5260	5390	5440
S3L-10	200	2950	3460	4040	90	4560	5070	5460	5540	5690

[a] Per cent by weight of mixing water; Sea-Rite added to sea water to obtain concentrations greater than 100 per cent.

[b] Three cylinders (4 in. in diameter by 8 in. long) for each age for each sea-water concentration except for 28-day age: 9 cylinders for each were used.

higher compressive strengths. Fresh concrete made without salt and fresh concrete made with mixing water having a salinity of 36.5 g per kg for the high water-cement ratio concrete showed almost identical air contents (5.2 and 5.0 per cent respectively) and almost identical unit weights (145 and 144 lb per cu ft respectively). In the hardened condition the unit weight of each of the concretes was 144 lb per cu ft. There was no significant bleeding of the concrete containing salt in the mixing water with a salinity of 36.5 g per kg.

The rather sharp, abrupt changes in the curves of Figs. 1(*a*) and (*b*) may be questioned; however, these changes are what the data indicate as the result of careful quality control in the fabrication and care of the concrete, which in turn is reflected by the low standard deviations for the 14-day strengths. Other natural phenomena showing such abrupt changes in relationships are not unknown.

Compressive strengths for the data in Table 2 may also be expressed by the following data model involving the various factors observed to affect strength:

$$S = 7929.7 + 863.7N - 1428.7N^2 + 326.2N^3 - 6327.2W/C + 1029.1T - 248.9NT + 12.1N\,W/C - 1031.3T\,W/C + 379.0NT\,W/C$$

where:

S = compressive strength, psi,

N = sodium chloride by weight of fresh concrete,
T = natural logarithm of age in days, and
W/C = water-cement ratio.

This data model was obtained by a least-squares fit to the data in Table 2. As an example of the meaning of this formula, consider a case where N is 0.6, T is 2.639 for 14 days age, and W/C is 0.444. For this combination of factors, S may be calculated to be 6577 psi, compared with the observed value of 6640 psi. Curves for this data model are compared with observed data plotted in Fig. 1(c).

The presentation of this data model is not a rejection of the peaked curves of Figs. 1(a) and (b). On the contrary, this model should be used only to approximate compressive strength as a function of the factors given and only within the range of the data and under the conditions specified for this experiment.

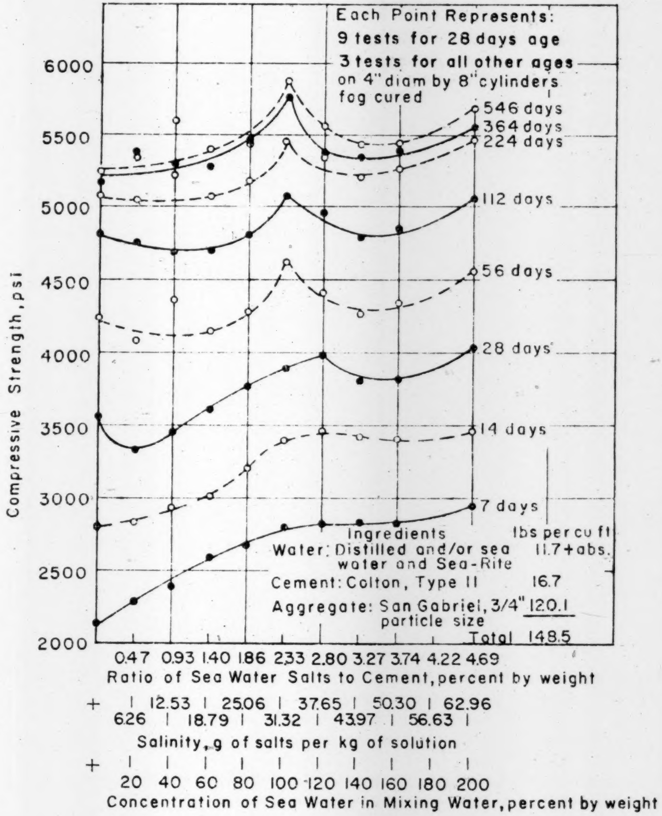

Fig. 2.—Compressive Strength Versus Sea-Water Concentration.

Sea Water Series:

The chemical composition of hypothetical sea water is given in Table 3.

On the basis of work by the International Commission (Torch, Knudson, and Sorenson, 1902), salinity is defined as the total amount of solid material in grams contained in one kilogram of sea water when all the carbonate has been converted to oxide, the bromine and iodine replaced by chlorine, and all organic matter completely oxidized (5).

Table 4 and Fig. 2 show compressive strengths of concrete versus different concentrations of sea water in the mixing water. Sea water from the NCEL sea water well, with a salinity of 31.32 g per kg (determined by chemical analysis), and distilled water were proportioned by weight. For example, in a 20 per cent sea-water concentration, 20 per cent of the mixing water by weight is sea water.

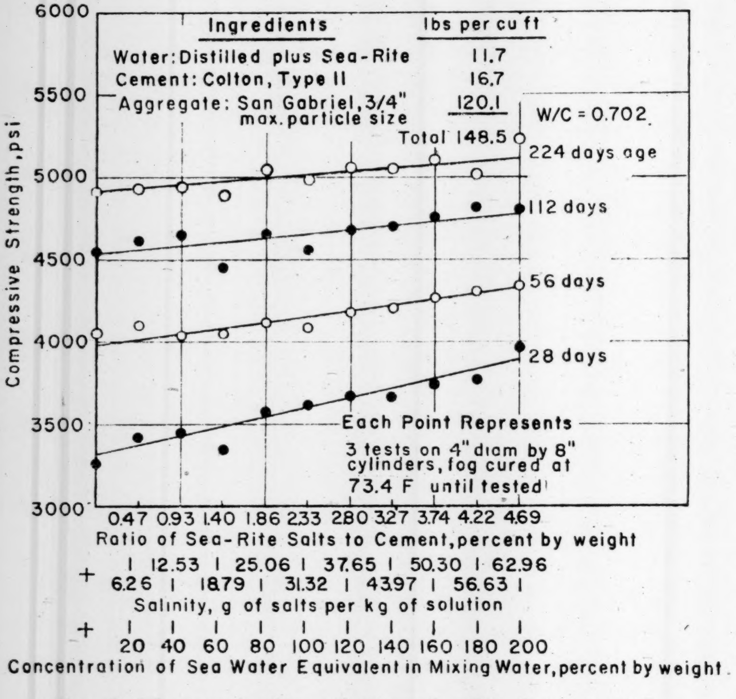

FIG. 3.—Compressive Strength Versus Sea-Rite Concentration.

Sea-Rite salt was added to sea water in order to obtain concentrations in excess of 100 per cent. Sea-Rite salt is a simulated sea-salt mix containing elements found in natural sea salt in quantities greater than 0.004 per cent. It is a granular, colorless salt.

Figure 2 shows a general increase in strength of concrete with age and with increases in concentration of sea water up to 200 per cent. In general, this figure shows that compressive strength continued to increase up to the limit of the test—546 days age. Unlike sodium chloride water, sea water had virtually no effect on slump of fresh concrete even with a salinity up to 63 g per kg.

The characteristics of the curves in Fig. 2 are somewhat unusual. In view of the rather pronounced characteristics of curves in Figs. 1(*a*) and (*b*), the authors believe there is a certain justification for the peculiar curves of Fig. 2. The low values for standard deviation in Table 4 lend some support to this belief. Moreover, the peak condition in

Fig. 2 occurs at nearly the same salinity as it does in Figs. 1(*a*) and (*b*). To determine if Sea-Rite salts alone in the mixing water would have the same effect, concrete cylinders were fabricated for strength determinations employing Sea-Rite salts in distilled water without the use of sea water. Figure 3 shows that the data are similar in magnitude and general trend to those in Fig. 2; however, the trends are much more nearly linear in Fig. 3.

Depth-of-Cover Series:

Concretes with two different water-cement ratios were fabricated for studies of water vapor transmission and steel corrosion. The compressive strengths of these concretes for various ages without additives or admixtures are as follows:

Batch	Water-Cement Ratio	Compressive Strength, 4 by 8-in. cylinders, psi						
		7 Days	14 Days	28 Days	56 Days	112 Days	224 Days	364 Days
S4L-2	0.702	2280	2820	3680[a]	4310	4890	5050	5200
S4H-2	0.444	4630	5630	6470[b]	7520	8070	8570	8950

[a] Standard deviation 28-day strength, 95 psi.

[b] Standard deviation 28-day strength, 175 psi.

NOTE.—Each value represents the average for three cylinders except for tests at 28 days, for which nine cylinders were used. Cement was Colton type II; average slump was 3 in. All cylinders were cured in 100 ± 2.0 per cent relative humidity at 73.4 ± 2.0 F.

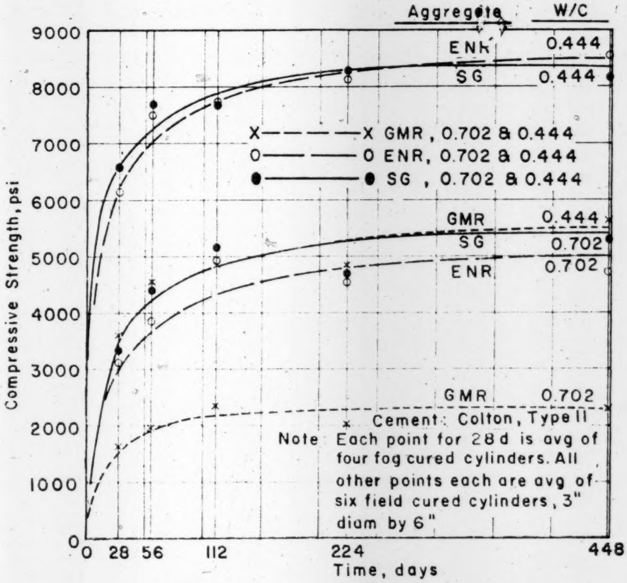

FIG. 4.—Compressive Strength Versus Age of Walls.

Wall Specimens:

Twelve small reinforced-concrete walls were cast using concrete described in Table 1 (described further under "Corrosion of Steel"). Figure 4 shows the results of compressive strength versus age for two water-cement ratios for each of three aggregates, the characteristics of which are presented in Table 1. Except for 28-day specimens, cylinders for compressive strength were field-cured

TABLE 5.—WATER VAPOR TRANSMISSION VERSUS SODIUM CHLORIDE CONCENTRATIONS IN MIXING WATER FOR WATER-CEMENT RATIO = 0.702.

(Also see Table 14)

Batch	Cup	Sodium Chloride Content[a]	*l*, in.[b]	*W/t*[c]	Water Vapor Transmission[d]	Water Vapor Transmission[d] Average
S2L-1..............	T483	0	1.992	0.625	0.370	0.375
S2L-1..............	T484	0	1.972	0.642	0.380	
S2L-2..............	T500	0.15	1.986	0.500	0.296	0.298
S2L-2..............	T501	0.15	1.902	0.505	0.299	
S2L-3..............	T547	0.30	1.987	0.504	0.298	0.295
S2L-3..............	T548	0.30	1.975	0.494	0.292	
S2L-4..............	T580	0.60	1.928	...	...	0.182
S2L-4..............	T581	0.60	2.160	0.308	0.182	
S2L-5..............	T597	1.20	1.983	0.291	0.172	0.176
S2L-5..............	T598	1.20	2.022	0.305	0.180	
S2L-6..............	T646	2.40	1.971	0.303	0.179	0.179
S2L-6..............	T647	2.40	1.986	0.301	0.178	

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[a] Sodium chloride content, per cent by weight of fresh concrete; Victor Type III cement.

[b] Length of flow path (or thickness of specimen).

[c] Slope of straight-line portion of graph of water loss in grams versus time in days; 520 days of record; 20 per cent relative humidity at 73.4 F.

[d] Grains per sq in. per day (1 g = 15.43 grains). Area of cross-section of flow path is 26.06 sq in. All specimens have a nominal diameter of 6 in.

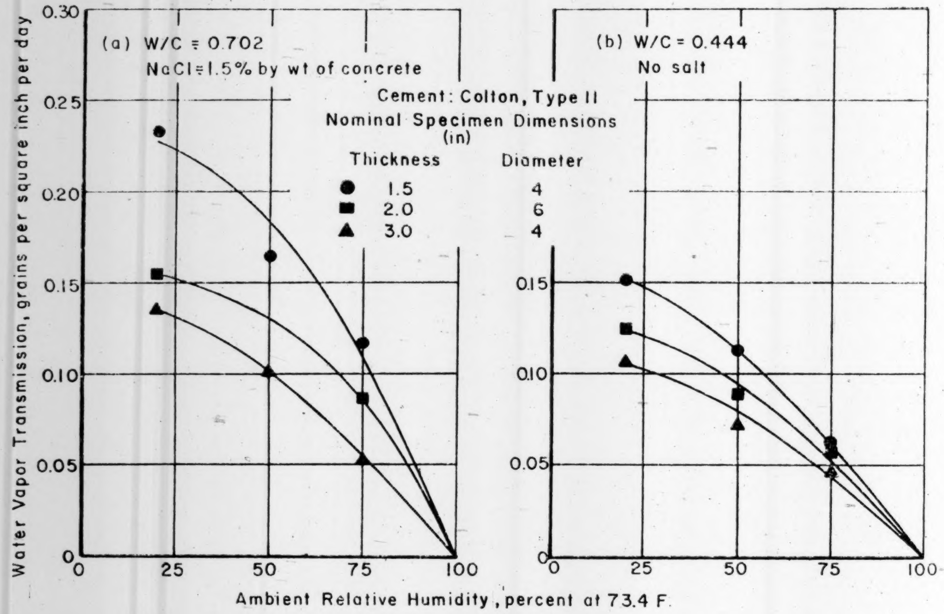


FIG. 5.—Water Vapor Transmission Versus Ambient Relative Humidity—With and Without Sodium Chloride.

on concrete pads on either side of the walls (land side and sea side). The values in Fig. 4 shown for the land side received no sea-water spray; the ones shown for the sea side received sea-water spray. Values for the sea side and land side generally were in such close agreement through 224 days age that average values were plotted in Fig. 4.

In general, for 448 days age, there are significant differences in compressive strengths for storage conditions (land side versus sea side). At this age, compressive strengths of cylinders receiving sea-water spray are all lower than corresponding cylinders protected from sea spray. For earlier ages, no strong pattern was established.

The effect of poor quality aggregate on strength of concrete is shown by a comparison of strength of concrete incorporating Guam reef coral (GMR) with the concrete incorporating superior aggregates such as San Gabriel (SG) and Eniwetok reef coral (ENR).

WATER VAPOR TRANSMISSION

Sodium Chloride Series:

Table 5 shows water vapor transmission (grains per square inch per day) values for concrete disks containing varying percentages of sodium chloride. If the data were plotted graphically, it would be observed that there is a rapid decrease in vapor transmission with increasing salinity of mixing water, reaching a minimum value at a salinity of about 70 g per kg; however, with further increasing salinity, the vapor transmission remains virtually constant.

Figure 5 is a plot of transmission values versus relative humidity for two water-cement ratios (0.702 with 1.5 per cent sodium chloride by weight of fresh concrete and 0.444 with no salt in the concrete) and for three thicknesses of specimen. This percentage of sodium chloride has significance because a large number of cores drilled from concrete structures on Pacific Ocean atolls contained chloride salts in this equivalent amount (1). For the structures sampled, there was no mix design information available, and it was therefore not possible to determine the amount of salts on a salinity basis or on a salt-cement ratio basis.

TABLE 6.—WATER VAPOR TRANSMISSION—DEPTH-OF-COVER SERIES (NO SALT).

Cup	Relative Humidity at 73.4 F, per cent	l, in.[a]	W/t[b]	Water Vapor Transmission[c]
S4L SERIES, WATER-CEMENT RATIO = 0.702, C-TYPE II CEMENT				
U744A.....	20	1.993	...	...
U741A.....	20	4.066	0.447	0.265
U740......	20	6.015	0.417	0.247
U750A.....	50	2.098	0.445	0.264
U747A.....	50	4.008	0.375	0.222
U750......	50	5.979	0.307	0.182
U746A.....	75	1.998	0.420	0.248
U745A.....	75	4.009	0.324	0.192
U740A.....	75	6.018	0.278	0.164
S4H SERIES, WATER-CEMENT RATIO = 0.444, C-TYPE II CEMENT				
U753A.....	20	2.056	0.204	0.121
U752A.....	20	3.995	0.138	0.082
U751......	20	6.041	0.144	0.085
U757A.....	50	2.020	0.164	0.097
U758A.....	50	4.020	0.095	0.056
U758......	50	6.004	0.086	0.051
U755A.....	75	2.002	0.120	0.071
U756A.....	75	4.033	0.060	0.036
U751A.....	75	5.998	0.048	0.028

[a] Length of flow path (or thickness of specimen).

[b] Slope of straight-line portion of graph of water loss in grams versus time in days; 400 days of record.

[c] Grains per sq in. per day (1 g = 15.43 grains). Area of cross-section of flow path is 26.06 sq in. All specimens have a nominal diameter of 6 in.

Comparison of Figs. 5(*a*) and (*b*) shows

TABLE 7.—WATER VAPOR TRANSMISSION VERSUS SEA-WATER CONCENTRATIONS IN MIXING WATER FOR WATER-CEMENT RATIO = 0.702.

NOTE.—Colton Type II cement.

Batch	Cup	Sea-Water Concentration[a]	Relative Humdity at 73.4 F, per cent	Nominal Diameter, in.[b]	l, in.[c]	W/t[d]	Water Vapor Transmission[e]
S3L-1	U306	0	20	6	2.000	0.488	0.289
S3L-2	U414	20	20	6	2.001	0.460	0.272
S3L-3	U447	40	20	6	2.006	...	...
S3L-4	U480	60	20	6	1.960	0.348	0.206
S3L-5	U514	80	20	6	1.996	0.302	0.179
S3L-6	U569	100	20	6	2.034	0.308	0.182
S3L-7	U605	120	20	6	2.009	0.303	0.179
S3L-8	U638	140	20	6	2.033	0.325	0.192
S3L-9	U671	160	20	6	2.043	0.313	0.185
S3L-10	U704	200	20	6	2.000	0.296	0.175
S3L-1	U307C	0	20	4	1.476	0.248	0.344
S3L-1	U307D	0	20	4	3.029	0.231	0.321
S3L-3	U448C	40	20	4	1.506	0.218	0.303
S3L-3	U448D	40	20	4	3.069	...	...
S3L-5	U515C	80	20	4	1.494	0.192	0.266
S3L-5	U515D	80	20	4	3.031	0.112	0.155
S3L-7	U606C	120	20	4	1.489	0.204	0.283
S3L-7	U606D	120	20	4	3.056	0.118	0.164
S3L-9	U672C	160	20	4	1.513	0.196	0.272
S3L-9	U672D	160	20	4	3.061	0.114	0.158
S3L-1	U307A	0	50	4	3.060	...	...
S3L-1	U307B	0	50	4	1.512	0.209	0.290
S3L-3	U448A	40	50	4	3.072	0.121	0.168
S3L-3	U448B	40	50	4	1.526	0.184	0.255
S3L-5	U515A	80	50	4	3.054	0.094	0.131
S3L-5	U515B	80	50	4	1.503	0.153	0.212
S3L-7	U606A	120	50	4	3.053	0.088	0.121
S3L-7	U606B	120	50	4	1.497	0.168	0.233
S3L-9	U672A	160	50	4	3.078	0.089	0.123
S3L-9	U672B	160	50	4	1.492	0.152	0.211
S3L-1	U308E	0	75	4	1.506	0.174	0.242
S3L-1	U308F	0	75	4	3.020	0.177	0.246
S3L-3	U449E	40	75	4	1.529	0.140	0.194
S3L-3	U449F	40	75	4	2.994	0.128	0.178
S3L-5	U516E	80	75	4	1.500	0.110	0.153
S3L-5	U516F	80	75	4	3.030	0.083	0.115
S3L-7	U607E	120	75	4	1.494	0.129	0.179
S3L-7	U607F	120	75	4	3.025	0.076	0.105
S3L-9	U673E	160	75	4	1.491	0.115	0.160
S3L-9	U673F	160	75	4	2.936	0.073	0.101

[a] Concentration of sea water in mixing water, per cent by weight.

[b] Area of cross-section of flow path for 6-in. cups is 26.06 sq in.; for 4-in. cups, area is 11.12 sq in.

[c] Length of flow path (or thickness of specimen).

[d] Slope of straight-line portion of graph of water loss in grams versus time in days; 440 days of record.

[e] Grains per sq in. per day (1 g = 15.43 grains).

that the effect of the salt in the low-strength concrete is to reduce its water vapor transmission values close to the level of that for the high-strength concrete without salt at the same respective ambient relative humidities.

Table 6 shows water vapor transmission values for specimens made in connection with a depth-of-cover corrosion study. If data from Table 6 (water-cement ratio = 0.444) were plotted and graphically compared with

Fig. 5(*b*), similar results would be observed. Data from Fig. 5 and Table 6 show slightly increasing rates of transmission with decreasing thicknesses of specimens. Table 6 also shows that transmission varies directly with water-cement ratio; that is, high water-cement-ratio concrete has higher transmission values than does low water-cement-ratio concrete.

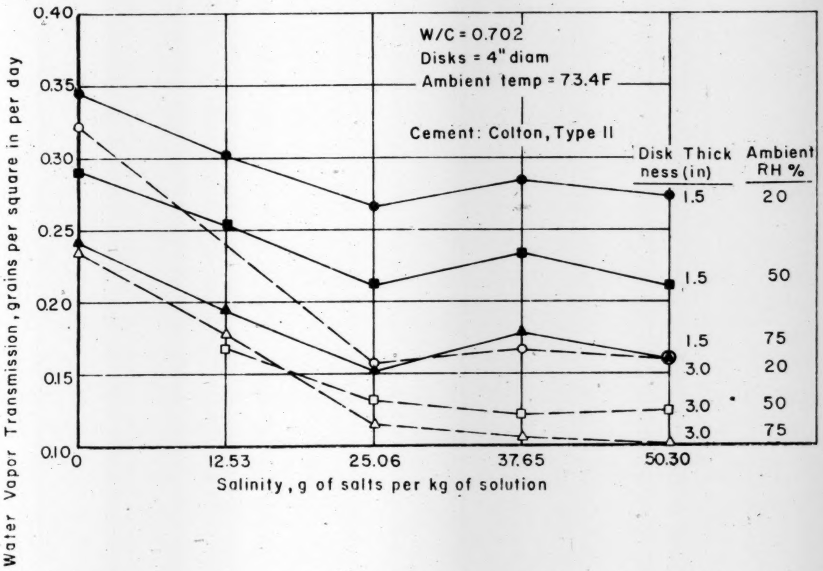


FIG. 6.—Water Vapor Transmission Versus Sea-Water Concentration—Thickness Effect.

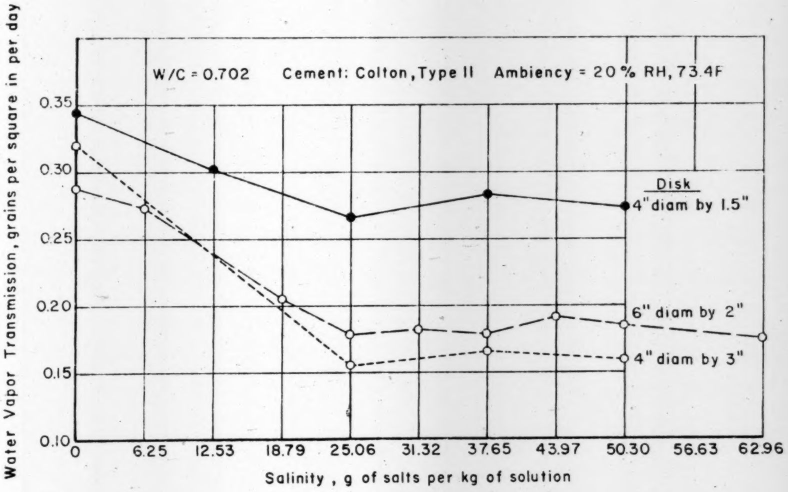


FIG. 7.—Water Vapor Transmission Versus Sea-Water Concentration—Diameter Effect.

Sea Water Series:

Transmission values for concrete disks containing varying percentages of sea water are shown in Table 7. Figure 6

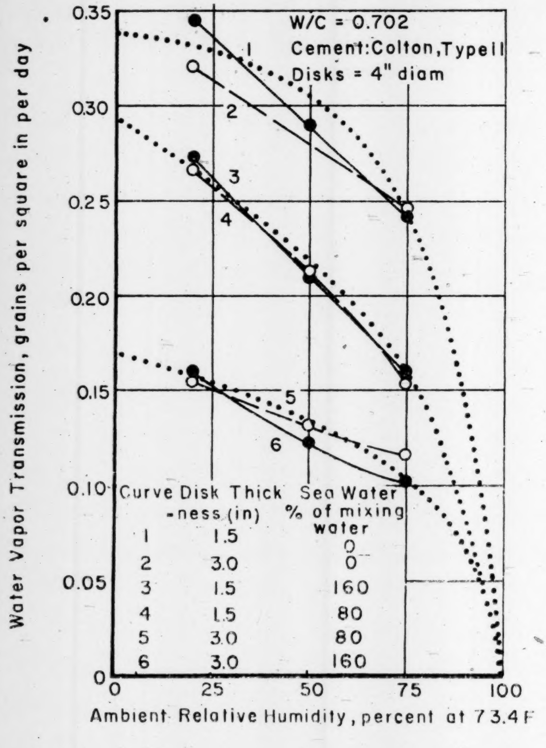


FIG. 8.—Water Vapor Transmission Versus Ambient Relative Humidity—With and Without Sea-Water.

TABLE 8.—WATER VAPOR TRANSMISSION OF KWAJALEIN CORAL CONCRETE.

NOTE.—Japanese Type II cement.

Cup	l, in.[a]	W/t[b]	Water Vapor Transmission[c]
S684M.......	2.017	0.374	0.221
S685M.......	1.995	0.376	0.223
S684T.......	4.081	0.203	0.120
S685B.......	4.098	0.224	0.133

[a] Length of flow path (or thickness of specimen).

[b] Slope of straight-line portion of graph of water loss in grams versus time in days; 570 days of record; 20 per cent relative humidity at 73.4 F.

[c] Grains per sq in. per day (1 g = 15.43 grains). Nominal diameter of cups is 6 in.; effective cross-section of flow path is 26.06 sq in.

shows variations in transmission versus salinity. If the data from Table 5 were plotted graphically and compared with Fig. 6, it might be observed that the nearly horizontal portion of the curves begins at a salinity of only 25.06 g per kg for sea water and of 70.5 g per kg for sodium chloride. Figure 6 also shows the effect of thickness of specimen. At zero salinity, the thicker specimens show only slightly lower transmission rates than do the thinner specimens; with increasing salinity, however, the thicker specimens show much lower transmission.

Figure 7 shows some limited evidence to indicate that specimens with larger cross-sectional areas have somewhat lower water vapor transmission (WVT) rates than similar specimens with smaller areas.

Figure 8 shows water vapor transmission as a function of ambient relative humidity. This figure shows that if no sea water is present in the mixing water, transmission is virtually independent of specimen thickness, and, if it is present, the thicker specimens show a greatly reduced transmission rate compared to the thinner specimens. The dotted lines are assumed to be reasonably correct.

TABLE 9.—WATER VAPOR TRANSMISSION OF WALL SPECIMENS.

NOTE.—Colton Type II cement.

Type of Aggregate[a]	Cup	l, in.[b]	W/t[c]	Water Vapor Transmission	Water Vapor Transmission[b] Average
			WATER-CEMENT RATIO = 0.444		
ENR	V84A	2.018	0.166	0.098	0.100
	V84B	2.031	0.170	0.101	
SG	U997A	2.080	0.212	0.126	0.135
	U997B	1.999	0.241	0.143	
GMR	V170A	1.994	0.453	0.268	0.272
	V170B	2.000	0.467	0.276	
			WATER-CEMENT RATIO = 0.702		
ENR	V41A	1.976	0.270	0.160	0.158
	V41B	2.055	0.264	0.156	
SG	U954A	2.021	0.492	0.291	0.302
	U954B	2.012	0.527	0.312	
GMR	V127A	2.004	0.580	0.343	0.335
	V127B	2.032	0.550	0.326	

[a] See Table 1.

[b] Length of flow path (or thickness of specimen).

[c] Slope of straight-line portion of graph of water loss in grams versus time in days; 360 days of record; 50 per cent relative humidity at 73.4 F.

[d] Grains per sq in. per day (1 g = 15.43 grains). Nominal diameter of cups is 6 in.; effective cross-section of flow path is 26.06 sq in.

Kwajalein Coral Concrete:

Table 8 shows WVT rates for two 6-in. diameter concrete cylinders made on Sept. 19, 1960. These cylinders were field-cured at Kwajalein for 24 hr at an average relative humidity of 73 per cent. They were then sealed in cardboard containers and delivered by air to NCEL on Sept. 26, 1960, and placed in a 100 per cent relative humidity, 73.4 F room until Oct. 17, 1960, when they were cut into disks and emplaced in wet cups in a 20 per cent relative humidity, 73.4 F room. A 2-in. thick disk and a 4-in. thick disk were cut from each of the cylinders. The pair of 2-in. specimens show remarkably close transmission rates; however, they are higher than any values obtained at NCEL for other concretes. Transmission rates for the 4-in. specimens agree reasonably closely and are in line with values for other concretes. Portions of ground concrete samples from each of the two specimens were dried, weighed, extracted in a Soxhlet apparatus for 96 hr, and analyzed spectrophotometrically for sodium chloride. The average sodium chloride content was found to be 0.09 per cent by weight of hardened concrete.

The coral aggregate was hard reef coral with a maximum particle size of 1.5 in. The aggregate was washed with fresh water to reduce the salt content to a minimum. The concrete incorporated an air-entraining agent and was made with Lion type II cement from Japan. The water-cement ratio was 0.554 and the slump was 3 in. Air entrainment was 3.5 per cent. The mixing water was fresh potable water.

Water Vapor Transmission of Wall Specimens:

Table 9 shows vapor transmission rates for specimens fabricated at the time the walls were cast. The specimens were fabricated from closely controlled batches in the pan-type mixer. Values for the SG aggregate specimens are slightly greater than for other NCEL specimens of the same aggregate. Values for the ENR high-quality coral aggregate specimens are the lowest of the three different aggregates. The GMR poor-quality coral aggregate specimens show distinctly higher WVT rates than either the ENR or SG, apparently reflecting its poor quality in high WVT values as well as in low comparative strength values.

Water Vapor Transmission Curve Characteristics:

Wet-cup specimens (disks) in this study were cut from fog-cured cylinders using a masonry saw. From the time the concrete was taken from the fog room until it was emplaced in a wet cup and stored in a constant-humidity room, not more than two days elapsed. Consequently, the initial weight loss of a wet cup was of the same nature as the weight loss of any saturated concrete specimen during drying in a constant relative humidity. As water evaporates from the specimen to some unknown value, the specimen begins to pick up water vapor from inside the cup and eventually transmits it out the drier side. Naylor (6) has shown that the water from the cup does not pass through concrete as a gas in any significant amount.

After some transitional period of water flow, a steady-state transmission rate of water is established which when plotted versus time was found to be a straight-line relationship. A very few specimens located in the 20 per cent as well as in the 75 per cent relative humidity (73.4 F) rooms exhibited a slightly curvilinear relationship in the so-called steady-state flow range, with the slope of the tangent to the curve decreasing with time. Almost all specimens in the 50 per cent relative humidity room in the present study showed this slightly curvilinear relationship. The reason for this is not completely clear. A new 50 per cent relative humidity room was used in this study, and one possible explanation is the precipitate on the surface of the specimens from the extremely hard water used to maintain humidity in this room. It is quite possible this substance has acted to lower the vapor transmission rates of the specimens in the 50 per cent relative humidity room in the same way that sodium chloride or sea water in the concrete did. The water vapor transmission graphs nearly all show that these values are lower than might be expected in relation to values for the 20 and 75 per cent relative humidity rooms. The transmission values for the 50 per cent relative humidity room were obtained by taking a chord of the relatively steady-state flow portion of each of the curves.

Asymptotically as time becomes quite

large, an empirical equation which well approximates the water loss of the wet cup (such as that illustrated later in Fig. 11) is:

$$w = a + bt$$

where:
w = weight loss of wet cup
a = intercept value on w-axis
b = slope of linear portion of curve
t = time
so that $b = dw/dt$ is the asymptotic WVT rate.

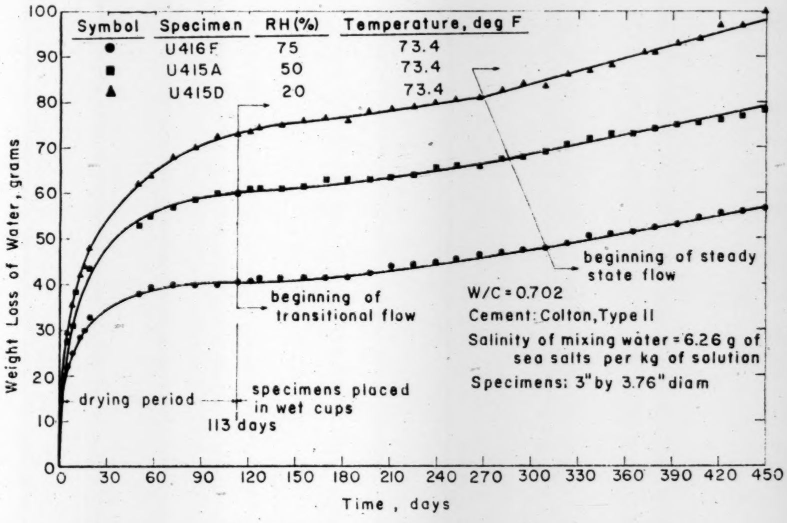


FIG. 9.—Effect of Drying Period on Water Vapor Transmission Rates.

In the previous study (1,2) water vapor transmission was defined by a formula which included the thickness of the specimen; however, this study shows that water vapor transmission is virtually independent of specimen thickness and therefore the thickness parameter was deleted giving the expression:

$$\text{WVT} = \frac{W}{At}$$

where:
W = weight of vapor transmitted, grains,
A = area of cross-section of specimen, sq in., and
t = time during which vapor transmission occurred, days.

Excluding vapor and ice as significant modes of moisture migration through concrete in the tests described herein, there remain four possible modes of moisture migration: chemical compound, absorbed, liquid, and dissolved. If the true mechanism of moisture migration could be determined, a great deal of understanding about the behavior of concrete would result.

Water Vapor Transmission Hysteresis:

Hysteresis is herein defined as a reduction in water vapor transmission caused by aging or a previous exposure of the specimen (7). For example, a number of specimens were first allowed to dry naturally in each of the three relative humidity rooms to nearly constant weight. These specimens, at equilibrium with their ambient environments, were then placed in wet cups, and water loss versus time was determined. Although insufficient time has passed to provide conclusive data, an example is shown in Fig. 9. To date the three specimens show transmission values of 0.128, 0.095, and 0.086 grains per sq in. per day respectively for 20, 50, and 75 per cent relative humidity. If these values are compared with Fig. 6, it may be noted that they are considerably lower than the curves of Fig. 6 indicate for a salinity of 6.26 g per kg.

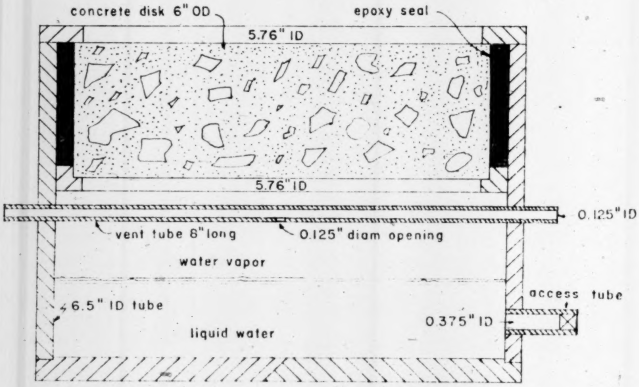

concrete disk 6" OD
epoxy seal
5.76" ID
5.76" ID
0.125" ID
vent tube 6" long
0.125" diam opening
water vapor
6.5" ID tube
liquid water
access tube
0.375" ID

FIG. 10.—Half-Section of Assembled Acrylic Wet Cup with Vent.

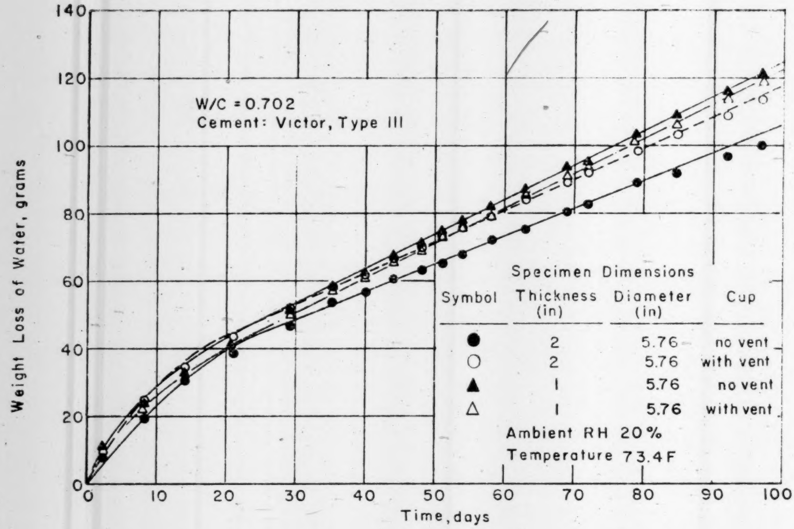


FIG. 11.—Effect of Vent in Wet Cup on Water Vapor Transmission.

Vented Cups:

With the exception noted below, all of the wet cups in the present as well as in the previous study (1,2) were unvented; that is, there was no opening directly into the vapor phase portion of the cup. Consequently, as water was transmitted from the cup through the concrete, partial vacuums may have developed and altered the vapor pressure.

According to Sears (8), a change in total pressure changes the vapor pressure. For example, an increase in the total pressure always increases the vapor pressure; if more of an inert gas is pumped into a container, more of the liquid in the container evaporates, contrary to what might be expected. The change in vapor pressure is very small compared with the change in total pressure.

In order to assure equal total pressures internal to and external to the cup, vents were installed in two cups as shown in Fig. 10. Companion specimens were placed in both vented and unvented cups. The curves of weight loss versus time for these four cups shown in Fig. 11 show that for the first 100 days there are no significant differences between the vented and unvented cups. Also, it shows no significant differences between specimens 1 and 2 in. in thickness. A control vented cup with acrylic top (instead of concrete) showed no significant weight loss. These cups were stored in 20 per cent relative humidity, at 73.4 F.

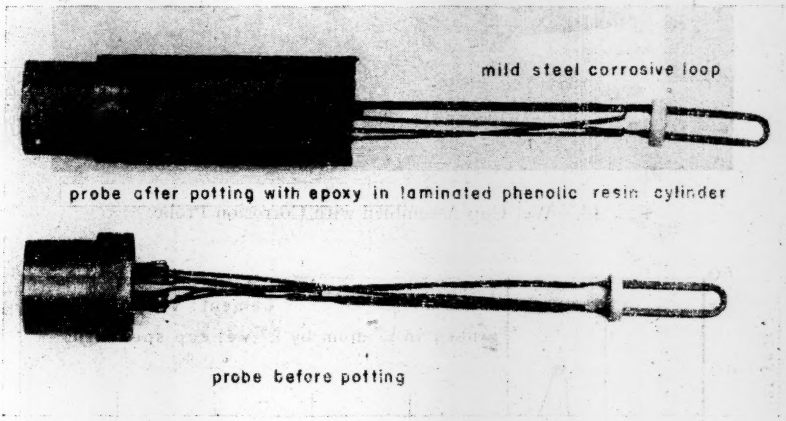

mild steel corrosive loop
probe after potting with epoxy in laminated phenolic resin cylinder
probe before potting

FIG. 12.—Electrical-Resistance Corrosion Rate Detection Probe.

CORROSION OF STEEL

Corrosion-Detection Probe:

The Corrosometer, the efficacy of which had been investigated previously (9), is an electrical-resistance corrosion-rate detection probe and metering device used to measure directly the degree of corrosive environment of reinforcing steel. Probes fabricated of mild steel of the type shown in Fig. 12 were obtained for this purpose. The phenolic-resin potting cylinder shown in this figure was made just long enough to extend about $\frac{1}{2}$ in. into the concrete. The probes were inserted into the fresh concrete through a hole in the wall of the cylindrical steel mold of the concrete cylinder. In order to do this, a cap was fastened across the top of the cylinder immediately after the concrete was cast; the cylinder was then laid on its side, the plug unscrewed from the hole, and the probe inserted. The cylinder was then replaced in its as-cast position and placed in the fog room. The exterior end of the probe was protected by a small rubber sheet fastened around it.

Corrosometer operation is based on the

fact that the electrical conductivity of most metals is very great compared with nonmetals. As the corrosion process converts metal into nonmetal, the electrical resistance of a piece of metal thus increases. This change of resistance is used to indicate the extent of penetration on an exposed metal specimen as corrosion proceeds on its surface.

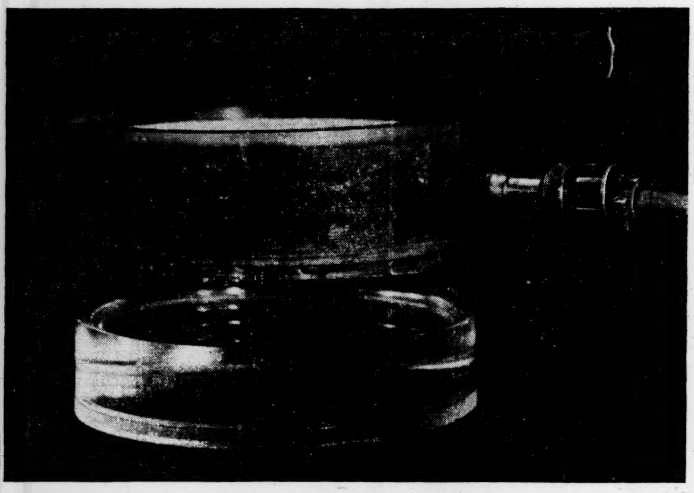


FIG. 13.—Wet Cup Assembled with Corrosion Probe.

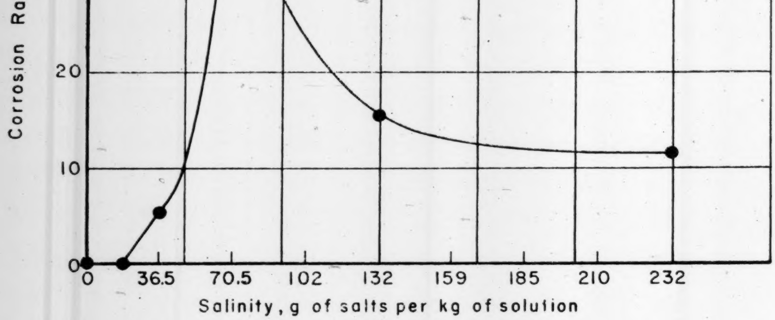


FIG. 14.—Corrosion Versus Salinity Using Sodium Chloride.

The resistance of this exposed specimen is not measured directly by the Corrosometer circuit. Instead, a second specimen made from the same metal or alloy is connected in series with the first and the two specimens are made part of a bridge circuit. The second specimen is covered with a highly corrosion-resistant coating to retain its original cross-section and resistance. The ratio of the resistance of the exposed specimen to that of the covered specimen is then determined.

By using a circuit which measures only the ratio of the two resistances, the measurements are made essentially independent of the current used to energize the bridge. Likewise, because temperature changes affect the resistance of both exposed and covered elements in the same manner, the resistance ratio and the meter reading are independent of the temperature of the environment. It is this self-compensating feature of the bridge arrangement that permits the high degree of precision and sensitivity attained by the Corrosometer.

Sodium Chloride Series:

Cylinders with probes were fabricated for concretes with different percentages of sodium chloride. Two-inch thick disks were cut with the probe in the middle and placed in wet cups as shown in Fig. 13. In order to place the disk with probe in an acrylic cup, a vertical strip was removed from the wall of the tube and cemented in place after the disk had been placed in the cup. The joint above the probe may be seen in Fig. 13. The cups were stored in 20 per cent relative humidity at 73.4 F.

Figure 14, giving results of the cups with concrete containing sodium chloride,

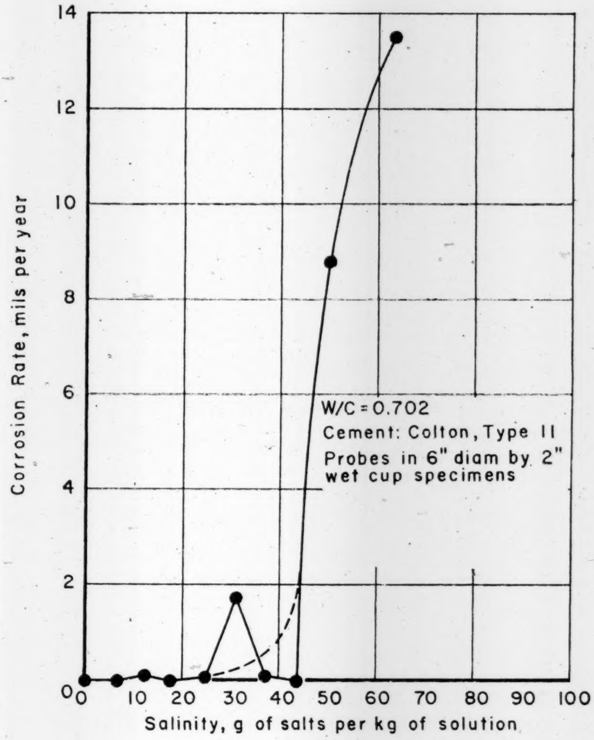


FIG. 15.—Corrosion Versus Salinity Using Sea Water.

TABLE 10.—VARIABLES FOR EXPERIMENTAL WALLS.

Statistical Factors	Wall											
	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7	No. 8	No. 9	No. 10	No. 11	No. 12
Aggregate[a]	GMR	SG	ENR	SG	ENR	GMR	ENR	SG	GMR	ENR	GMR	SG
Strength[b]	Hi	Lo	Lo	Hi	Hi	Lo	Hi	Lo	Lo	Lo	Hi	Hi
Steel arrangement[c]	T	W	T	W	T	W	W	T	T	W	W	T
Depth of steel (cover), in.	1	1	6	6	1	1	6	6	6	1	6	1
Wall thickness, in.	3½	3½	13½	13½	3½	3½	13½	13½	13½	3½	13½	3½
Casting date (1961)	May 15	May 8	May 11	May 8	May 11	May 15	May 11	May 8	May 15	May 11	May 15	May 8
Cylinder test dates.	July 10, 1961	July 3, 1961	July 6, 1961	July 3, 1961	July 6, 1961	July 10, 1961	Aug. 2, 1962	July 30, 1962	Aug. 6, 1962	Aug. 2, 1962	Aug. 6, 1962	July 30, 1962
	Sept. 5, 1961	Aug. 28, 1961	Aug. 31, 1961	Aug. 28, 1961	Aug. 31, 1961	Sept. 5, 1961	May 11, 1963	May 8, 1963	May 15, 1963	May 11, 1963	May 15, 1963	May 8, 1963
	Dec. 26, 1961	Dec. 18, 1961	Dec. 21, 1961	Dec. 18, 1961	Dec. 21, 1961	Dec. 26, 1961	May 11, 1964	May 8, 1964	May 15, 1964	May 11, 1964	May 15, 1964	May 8, 1964

[a] See Table 1.

[b] Hi = High; water-cement ratio = 0.444;
Lo = Low; water-cement ratio = 0.702.

[c] T = Tied (and steel contacts insulated);
W = Welded.

shows that a salinity of 70.5 g per kg is most favorable for the maximum rate of corrosion. The pattern of corrosion versus salinity is very similar to that shown on page 131 of reference (10).

Sea Water Series:

A similar set of concrete disks incorporating varying percentages of sea water in the mixing water was also prepared and placed in wet cups stored in 20 per cent relative humidity at 73.4 F. Figure 15, showing the corrosion rates for these probes, indicates that no significant corrosion occurs up to a salinity of 25 g per kg; thereafter, the corrosion rate increases markedly. The salinity in seawater solutions for the maximum corrosion rate is evidently beyond 63 g per kg (200 per cent sea water).

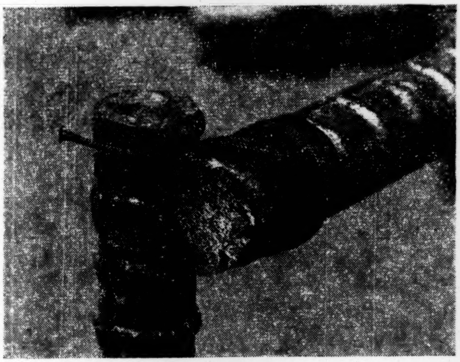


FIG. 16.—Details of Steel Insulation.

Depth-of-Cover Series:

A series of 6-in. diameter by 6-in. thick concrete specimens for each of two different water-cement ratios (0.444 and 0.702) was prepared with corrosion-detection probes. No salt was used in the concrete. The specimens were placed in wet cups and stored in 50 per cent relative humidity at 73.4 F. Measuring from the bottom surface of the specimen, the probes had depths of concrete cover of 1, 2, 3, 4, and 5 in. A single probe was placed in each concrete disk, and each concrete disk was made in duplicate (each series had 10 cups). A time period of 400 days has shown no measurable corrosion in any of the 20 cups involved. Water transmission values of these cups are represented by cup No. U750 and cup No. U758 in Table 6.

Experimental Walls:

In order to simulate a marine environmental exposure such as that encountered by a building on a tropic atoll, small walls of reinforced concrete were built and seawater spray was applied daily. The details of the concrete mixes and their ingredients are shown in Table 1.

The variables to be investigated are shown in Table 10. The three types of aggregate described in Table 1 and two water-cement ratios (0.444 and 0.702) were used, and two different reinforcing-steel arrangements were employed. Each of the steel arrangements had the same grid spacings. The only difference in arrangement was that in one case the mild-steel bars were insulated from each other with electricians' plastic tape and tied together with nylon fishing cord, as shown in detail in Fig. 16. In the other arrangement all steel bars were tack-welded together at the point of contact. These arrangements were to permit the investigation of corrosion of individual steel bars as compared to the corrosion of an interconnected grid of steel bars. Two depths of steel cover were also included as variables, the depth being measured from the outer surface of the concrete to the nearest surface of the steel. It was desired to eliminate all external entries, such as tie-wire and bolt holes, through the concrete to the steel as unknown variables; therefore concrete blocks for the particular kind of concrete to encase the steel grids were precast with wire loops embedded in each

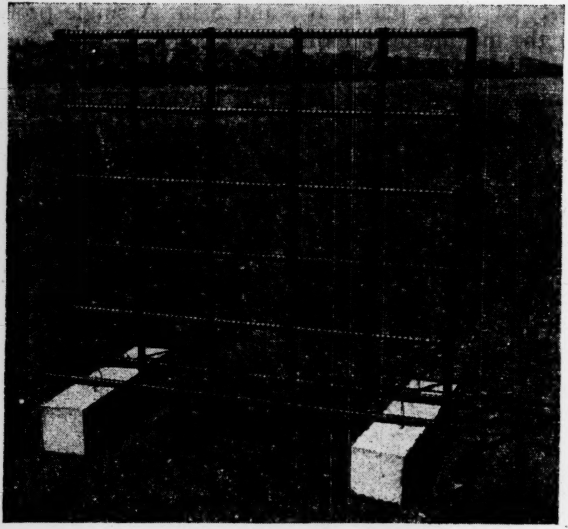


FIG. 17.—Concrete Supports for Steel Grids.

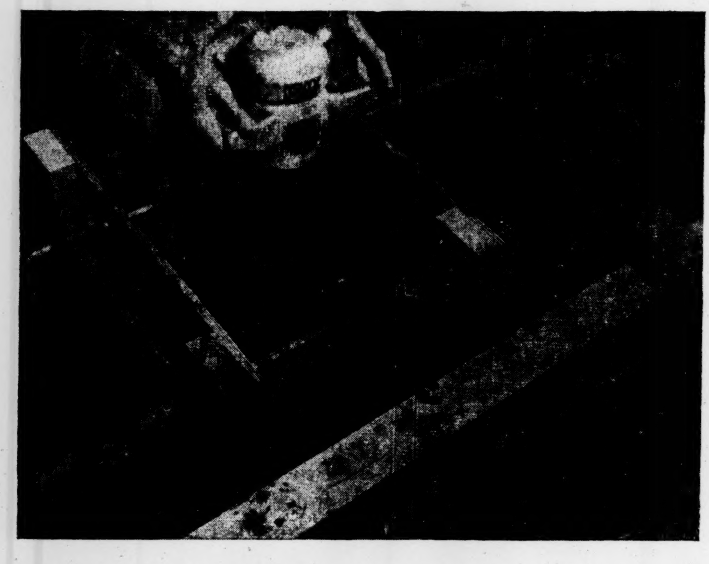


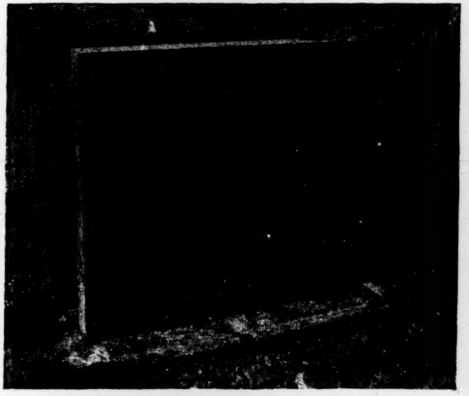
FIG. 18.—Temporary Devices for Holding Steel Grids in Forms.

block for securing the steel grids (Fig. 17). In this way there were no steel wires going from the steel grid through the concrete to the outside. The use of spacer blocks was avoided for the same reason, and the grids were held in place vertically by means of temporary holding devices which may be seen in Fig. 18.

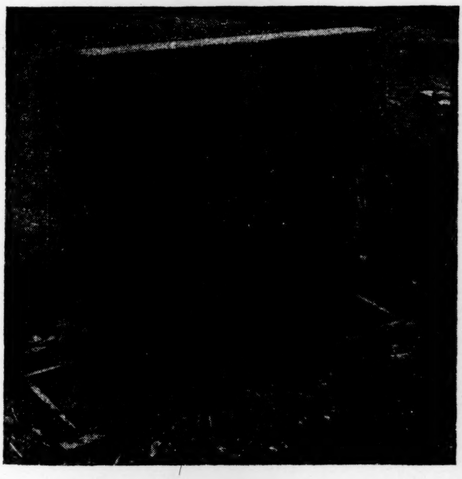
It was desired to hold the water-

TABLE 11.—SLUMPS FOR CONCRETES USED IN WALLS.

Wall	Thickness, in.			Water-Cement Ratio	Aggregate
	Minimum	Average	Maximum		
6, 9	4½	6½	8	0.702	GMR
2, 8	1	2	3	0.702	SG
3, 10	1½	5	6½	0.702	ENR
1, 11	½	1½	3	0.444	GMR
4, 12	3	3½	4½	0.444	SG
5, 7	3	4½	6	0.444	ENR



(*a*) San Gabriel aggregate concrete wall.



(*b*) Coral concrete wall.

FIG. 19.—Comparison of Concrete Walls After Stripping Forms.

cement ratio for the high-strength concretes equal throughout and that for the low-strength concretes equal throughout. It was further desired to design all concretes for identical consistencies as measured by the slump test. This required a greater amount of water and cement for the coral aggregate concretes than for the San Gabriel aggregate concrete.

While the slump test has for a number of years appeared to be an excellent measure of consistency for SG aggregate concrete, coral aggregate concretes do not respond quite so uniformly to the slump test. Although distribution of aggregate by percentage was approximately the same for all batches, the coral concretes appeared too harsh, compared with the SG aggregate concrete. The slumps in inches for the various concretes used in the walls are given in Table 11.

The apparent harshness of the coral concretes was further demonstrated in the construction of walls when the forms were stripped at 7 days age (Figs. 19(*a*) and (*b*)). Although the apparent consistencies were the same, the coral concretes did not flow down into the foundation of the wall as well as did the SG concrete. This occurred even though the stud vibrator was applied internally to the concrete as well as externally to the form.

During the first seven days of curing, the forms of each wall remained in place, and burlap placed across the top of the walls was kept wet by means of water spray. On the seventh day, after the forms were stripped from each wall the voids, as shown in Fig. 19(*b*), were filled in with appropriately designed fresh concrete. On the eighth day each wall was rubbed down with mortar employing the same fine sand that had been used in the concrete mix for each particular wall. The walls were then completely wrapped in burlap, which was kept soaking wet. On the 28th day after placing, the walls were stripped of burlap, and 3-in. diameter by 6-in. concrete cylinders for determining compressive strength were placed along the walls as shown in Fig. 20. When the last walls to be cast (GMR) had achieved an age of 30 days, the first application of sea water was made to the seaward side of each wall on June 15, 1961, for a 5-min interval once each day. The sea water was obtained from the NCEL sea-water well.

When the GMR walls were 29 days old, the first set of monthly photographs was taken for the purpose of a statistical evaluation of time-dependent deterioration. On July 7, 1961, considerable efflorescence was noted on walls 6, 9, and 11. During the months of January, February, and March, 1963 (exact dates were not determined) significant vertical cracks in the end faces of walls 1, 2, and 10 occurred.

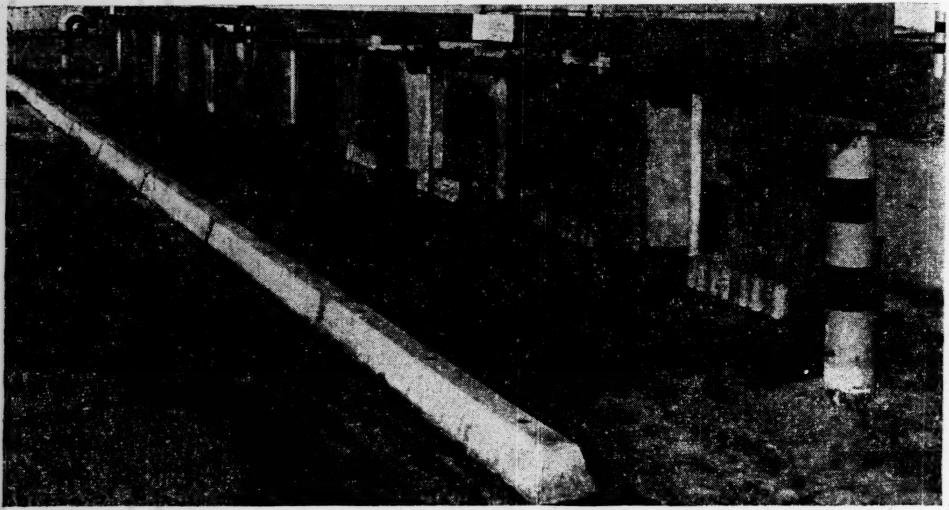


FIG. 20.—Over-all View of Experimental Walls.

GENERAL DISCUSSION

Strength:

A contributing factor to deterioration of concrete structures on tropical atolls is believed to be the presence of foreign salt. When hardened concrete is analyzed chemically, the chloride content is usually expressed in combination with sodium. For this reason, the effect of sodium chloride was of interest. The actual combination of chloride ions with other metals in concrete is very difficult to estimate accurately.

In a marine environment, sea water is the source of a significant amount of foreign salt found in concrete. There may be sea-water salts in the aggregate, and sea water may be used in the mixing water. Also, on atolls, sea spray on concrete structures continually adds salt to the hardened concrete by penetration. A satisfactory external water-repellant coating on the concrete would prevent sea-spray salts from penetrating the concrete.

For the average maximum of 1.5 per cent sodium chloride by weight of hardened concrete found in tropical concrete, the corresponding salinity for concretes in this study is 159 g per kg. Roughly this would be equivalent to the salts in 4 kg of sea water reduced to 1 kg of saline solution. It is very difficult to see how this concentration of salt could get into the concrete at the time of mixing; however, a concrete structure exposed to sea spray might accumulate this concentration of salts over a period of years. Since the latter can be prevented, interest is mainly in the amount of salt permissible at the time of mixing.

Neither sea water with a salinity up to 63 g per kg nor sodium chloride water with a salinity up to 36 g per kg have adverse effect on compressive strength of concrete up to one year as determined in this study. The general conclusion of Lea and Watkins (11), emerging from their study of reinforced-concrete piles exposed to sea-water action for up to 23 years, was that the primary cause of deterioration of the piles was corrosion of the reinforcement and not the disintegration of the concrete from chemical or physical effects of the salts in the sea water. The senior author (Griffin) of the present study, from his own observations in the islands of Canton, Eniwetok, Kwajalein, and Midway, found no reason to disagree with this thesis.

Lea outlines prime essentials for sea-water concrete on pages 552 and 553 of reference (12). He states that sea water may be used for mixing plain concrete for sea-water structures except for high-alumina cement. High-alumina cement concrete made with fresh water, however, has greater durability when exposed to sea water than does portland cement. The durability of pozzolanic cements was lower than for high-alumina cement and higher than for portland cement under the same conditions (11).

Water Vapor Transmission:

The use of lower water-cement ratios decreases rate of water vapor transmission. The use of sodium chloride water with salinities up to 70 g per kg and of sea water with salinities up to 25 g per kg decreases transmission values.

Corrosion of Steel:

For either sodium chloride water or sea water in concrete, salinities up to 25 g per kg do not produce highly significant corrosion rates. For sodium chloride water, salinities in excess of about 160 g per kg do not produce highly significant corrosion rates. Salinities between 25 g per kg and 160 g per kg of sodium chloride solutions in concrete do produce highly significant corrosion rates. This pattern may be true for sea-water concentrations also; however, the full pattern was not developed in this study.

The experimental walls should reveal the extent of salt gain from sea-water spray and the length of time for corrosion of the reinforcing steel to manifest itself either by an appearance of rust stains or by cracking of concrete caused by a formation of corrosion products on the reinforcing rods.

Summary:

It is apparent that some salt is beneficial to reinforced concrete. Concrete with mixing water with a salinity of 25 g per kg, corresponding to 80 per cent sea water and 20 per cent fresh water by weight, shows a gain in strength, lower values for water vapor transmission, and no significant corrosion of mild steel.

CONCLUSIONS

Compressive Strength:

1. Concrete incorporating sodium chloride in the mixing water shows an optimum salinity of about 36 g per kg for maximum compressive strength.

2. Depending upon age of concrete, relatively high concentrations of sodium chloride are permissible before strengths less than those for zero salinity result.

3. Natural sea water does not deteriorate plain concrete nor does it cause any

reduction in strength. On the contrary, strength is generally increased with up to double the normal concentration of salts in sea water.

Water Vapor Transmission:

1. With increasing sodium chloride salinity of mixing water up to 70 g per kg and of sea-water salinity up to 25 g per kg, water vapor transmission is reduced to its minimum. With further increasing salinities, vapor transmission is almost constant.

2. With increasing salinities, the effect of the thickness of the specimen becomes much more pronounced, with thicker specimens showing much greater reductions in vapor transmission than do thinner specimens.

3. With or without salts, vapor transmission varies directly with the water-cement ratio. Concretes with higher water-cement ratios have higher transmission values.

4. Without salts, thinner concrete specimens have somewhat higher vapor transmission rates than do thicker specimens. Indications are that 6 in. may be a limiting thickness beyond which greater thicknesses do not further decrease WVT significantly.

5. Concrete made with poor-quality aggregate (GMR) shows higher transmission values than concrete made with high-quality aggregate (SG and ENR), other factors being equal.

6. The present study does not confirm nor—probably because of the situation described for the 50 per cent relative humidity room—does it completely negate an earlier finding (1) that water vapor transmission is independent of ambient relative humidity (of 20 or 50 per cent at 73.4 F).

7. No mathematical relationship could be established to predict water vapor transmission for specimens of identical concrete but with different thicknesses.

Corrosion:

1. The electrical-resistance detection probe appears to be a reliable device for measuring the corrosiveness of concrete to reinforcing steel.

2. Concrete incorporating sodium chloride in the mixing water shows an optimum salinity of 70 g per kg for maximum corrosion of steel.

3. Sea water with a salinity of 25 g per kg or less in the concrete mixing water does not significantly corrode steel.

General:

A small amount of sea-water salts is beneficial to concrete. At a mixing-water salinity of 25 g per kg, strength is improved, water vapor transmission is minimized, and corrosion of mild steel is not significant.

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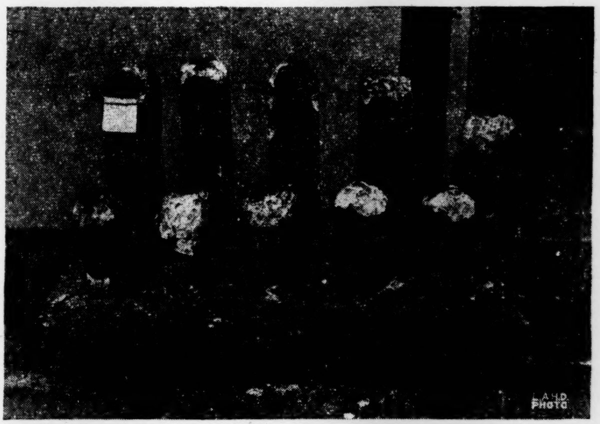
DISCUSSION

MR. C. M. WAKEMAN[1] (presented in written form).—The authors are to be congratulated for their carefully planned research in a field which has been neglected far too long. Their presentation points out a new and interesting approach for future investigations.

Presentation of data to indicate the relationship of water vapor transmission to that of relative humidity is particularly interesting. These studies may indicate an explanation for failure in one part of a concrete structure and not in another. For example, greater deterioration was found on that portion of the San Mateo Bridge structure[2] which was constantly damp than in the portion subjected to the drying action of prevailing winds.

The authors state in their summary



All specimens showed advanced deterioration by softening and spalling of the concrete.

FIG. 21.—A Collection of Unreinforced, 6 by 12-in. Concrete Cylinders and 6 by 6 by 36-in. Beams After Being Submerged in Clean Sea Water for Ten Years.

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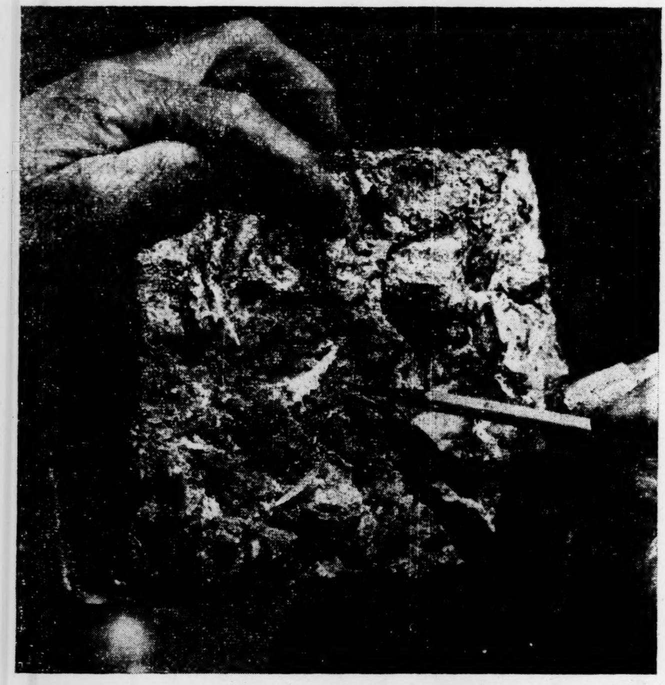
[1] Testing Engineer, Port of Los Angeles, Wilmington, Calif.

[2] R. F. Stratfull, "Corrosion of Reinforcing Steel in Concrete," *Corrosion*, June, 1959, p. 65.

that, "... some salt is beneficial to reinforced concrete." It is obvious that this assertion needs an explanation, or at least a modification to the effect that their statement was true only insofar as the confines of their test was concerned. I am sure that they did not intend this to be a general statement that chlorides should be deliberately included as an ingredient of all marine concrete. Furthermore, if the period of their test had been extended ten years instead of one, perhaps this statement could not have been made at all.

Conclusion 3 (Compressive Strength) is probably also true only under the specific conditions of their tests and likewise should not be construed as a general statement. There is ample evidence[3] that unreinforced concrete of low cement content, porous structure, or both, can and does deteriorate in strength when exposed to sea water (see Figs. 21 and 22).

If the photograph of the concrete wall (the authors' Fig. 19(*a*)) is typical of all of the panels, it is difficult to reconcile the large number of surface imperfections (water pockets) with proper internal and external vibration during their casting. Furthermore, in the opinion of the writer, the practice of filling these voids with "plastic concrete" followed by a rubdown with mortar, is not a good substitute for a perfect finish. While this procedure is not an uncommon practice in building construction, results merely serve to make the completed panels more pleasing to the eye, but they provide little aid to durability.



The concrete had suffered deterioration by softening and spalling.

FIG. 22.—Close-up View of One of the Concrete Beams Shown in Fig. 21 After Ten Years Exposure to Clean Sea Water.

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[3] Unpublished report from the Port of Los Angeles Testing Laboratory.

Mr. D. F. Griffin (*author*).—I am sure most people recognize that conclusions drawn out of test data are, of course, based specifically on those test data, and that to extend the conclusions to other situations is only an estimate of what might happen. At the moment I do not recommend that any salt be added to the fresh concrete of a structure to be exposed in a marine environment since we do not yet have an economically feasible method for preventing accumulation of salts in concrete exposed to sea mist and sea spray. However, I would be less than truthful if I were not willing to recognize the validity of my own test data. In a nonmarine environment, based on our findings, I would recommend a small amount of sodium chloride as being beneficial. Even in this case I would not recommend it unless a very careful control could be established. I think that answers based on 10 yr of experimentation are certainly superior to answers based on a shorter term of investigation; however, research is for the purpose of making future estimates; therefore we have to use the best estimates available as we go along.

With reference to conclusion 3 on compressive strength, again conclusions are based only on the data shown in our report. We would certainly expect that concrete of very low cement content, particularly if it is porous, would deteriorate in sea water. I am sure there is a relationship between ability of concrete to maintain its integrity in sea water versus its permeability. For example, some experimental piles at the Portsmouth Naval Shipyard were recently evaluated after some 35 years of exposure. There were two items of primary interest in connection with this:

1. A general appraisal of the piles in the category of "good," "fair," and "poor" showed a direct correlation with water vapor transmission values determined for cores taken from these piles, the good piles showing low water vapor transmission values and the poor piles showing high rates of water vapor transmission. There was no direct correlation between these ratings and the sodium chloride content of the piles.

2. A second item of interest is the fact that most of these piles began to deteriorate not in the immersed portion of the pile but in the tidal portion of the pile. In this zone of alternate wetting and drying, sea salts could certainly accumulate at a fairly rapid rate. Within the immersed portion of the piles the salinity would always be in balance with the sea water and not exceeded. As shown by Figs. 1(*a*) and (*b*), the higher salt contents in concrete have a weakening effect.

The photograph of the concrete wall in Fig. 19(*a*) is typical of the wall panels. The number of surface imperfections for this particular concrete mix design is not inconsistent with results for similar concrete we have encountered with slumps of 3 in. or less. The reason for filling the surface voids with mortar was simply to eliminate surface water pockets and provide a uniform type of surface for all panels.

Mr. Bailey Tremper.[4]—What is the effect of concentration of salt by evaporation on corrosion of steel?

Mr. R. L. Henry (*author*).—The effect is described by the entirety of the report and specifically by Figs. 14 and 15 of this paper.

Mr. W. H. Price.[5]—Could 25,000 ppm of sodium chloride safely be used in concrete without causing corrosion of reinforcement steel?

Mr. Henry.—Within the limits of the findings of this experiment, 25,000 ppm of sodium chloride could be used without causing significant corrosion, as can be seen in Fig. 14 of this paper.

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[4] California Portland Cement Co., Riverside Calif.

[5] Riverside Cement Co., Los Angeles, Calif.

MR. P. E. McCOY.[6]—Standard Oil of California built a long wharf in San Francisco Bay about 16 years ago. In order to protect the piling and decks from scaling, Standard Oil elected to include Hydropel, an asphalt emulsion (low pH type), made for use with concrete as an integral waterproofing admixture. About a year ago a series of plain and Hydropel admixture treated concrete specimens were removed from the bay for examination. Some of these contained reinforcing bars completely embedded in the concrete. Specimens were broken apart and the bars carefully examined. Those from the plain concrete were severely corroded. Rods from the Hydropel admixture concrete were completely free from corrosion.

Evidently, the Hydropel admixture, by retarding the intrusion of salt water over the past 16 years, keeps the salt concentration below the critical point at which corrosion of the reinforcing bars occurs. It offers a solution for the prevailing problem of corrosion of reinforcement in marine atmospheres.

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[6] **Assistant to Vice-President-Technical, American Bitumuls and Asphalt Co., San Francisco, Calif.**

ACCELERATED STRENGTH TESTING OF CONCRETE CYLINDERS*

By P. Smith[1] and B. Chojnacki[1]

Synopsis

The 7 or 28 days required by the present standard procedures for curing and testing cylinders in compression is often too long a period to wait to learn whether the strength of the concrete is satisfactory. A laboratory program and field trials have therefore been undertaken to develop an accelerated procedure for curing test cylinders so that results can be available on the day following the placing of the concrete. The provisional procedure consists of placing the cylinders in boiling water 20 min after the concrete reaches a pin pull-out bond strength of 12 psi or Proctor needle penetration resistance of 3500 psi. After the cylinders are placed in the water, it is again brought to boiling in 2 hr. After 16 hr of curing in water the cylinders are removed from the boiling water and tested in compression within 1 hr.

Since the delay time before the cylinders are placed in the boiling water is based on the degree of set of the concrete, variations due to differences in cements and admixtures are allowed for. For the materials and variables tested, the relationship of accelerated (R_a) to 28-day (R_{28}) compressive strength is $R_{28} = (26160\ R_a)/(R_a + 11620)$.

Consideration is given to the value of accelerated strengths as the basis for rapid quality control in the field. Since the 28-day strength is an arbitrary measure of the quality of the concrete, a suitable accelerated curing procedure could offer a more convenient and realistic way of ascertaining if the concrete will satisfy the purpose for which it was designed.

Although it is now 30 years since Patch (1)[2] used accelerated curing of concrete cylinders to obtain early estimates of the strength of the concrete used on the Hoover Dam, full advantage does not appear to have been taken of his idea. It is still common practice to carry on with the next stage of construction before adequate confirmation that the concrete meets the compressive strength requirements of the specification, which are usually stated in terms of the strength of cylinders that have received 28 days of standard curing.

The need for earlier information has conventionally been met by taking 7-day compressive strength results as a first check and as a base from which to predict the eventual 28-day strengths. At best this is only a partial answer since the 7- to 28-day strength relationship may be imprecise and the time of testing is still too long for the results to be of real practical value.

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* Presented at the Sixty-sixth Annual Meeting of the Society, June 23-28, 1963.

[1] Senior Materials Engineer (Concrete) and Concrete Development Engineer, respectively, Materials and Research Division, Department of Highways—Ontario, Toronto, Ont.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

Faced with the laying of concrete pavement at the rate of one mile per day, the Materials and Research Division of the Department of Highways, Ontario, undertook to see if accelerated test methods yielding results by the following day could be developed for such properties as concrete strength, compaction, and riding quality (2). This paper is concerned with the development of an accelerated curing procedure which could eliminate waiting to find out whether the compressive strength of the concrete is satisfactory.

TABLE 1.—PHYSICAL AND CHEMICAL TESTS ON CEMENTS (STANDARD DELAY ACCELERATED CURING PROCEDURE).

CEMENT SOURCE & BATCH Nº.	LAB. SAMPLE Nº.	PHYSICAL TESTS						CHEMICAL TESTS						
			FINENESS											
		NORMAL CONSISTENCY %	RET. #200 %	BLAINE $cm^2/gm.$	AUTOCLAVE EXPANSION %	VICAT INITIAL SET FINAL SET hr.-min.	COMP. STRENGTH (psi) 3 DAY 7 DAY 28 DAY	LOSS ON IGNITION %	INSOLUBLE RESIDUE %	TRICALCIUM ALUMINATE %	SULPHUR TRIOXIDE %	MAGNESIA %	SODIUM OXIDE %	POTASSIUM OXIDE %
PRELIMINARY ACCELERATED CURING PROCEDURE														
1-1	60-S 56805	24.0	2.8	–	0.04	2-50 4-30	2442 3092 4342	1.17	0.30	2.20	2.20	2.82	0.66	0.54
1-2	61-S 56803	24.0	1.4	–	0.04	3-00 5-20	2383 3158 5225	0.70	0.07	4.82	2.23	1.61	0.60	0.69
2-1	61-S 56801	24.5	4.2	–	0.44	3-25 5-15	2367 3283 5100	1.39	0.07	10.96	2.52	1.60	0.25	0.98
3-1	61-S 56802	24.0	6.0	–	0.19	3-20 5-30	1850 2909 5050	1.48	0.09	7.98	2.34	2.77	0.41	0.65
4-1	61-S 56804	24.5	0.6	–	0.25	4-15 6-05	2350 3900 5792	1.04	0.32	7.34	2.27	1.62	0.50	0.54
5-1	61-S 56805	24.5	4.6	–	0.04	2-40 4-45	2583 3458 4742	0.73	0.19	12.36	2.57	0.86	0.53	0.92

When the project started in 1958, reports of recent work in England on the rapid curing of concrete test cubes indicated that strength development could be accelerated by raising the curing temperature. King (3,4) used dry heat in an oven while Akroyd and Smith-Gander (5) used hot water. A review of their findings and some preliminary experiments led to the selection of hot or boiling water as the accelerating medium since this appeared to offer the best prospects of uniformity and reproducibility with simple equipment.

Laboratory tests were undertaken to determine a suitable delay time before cylinders were placed in the hot or boiling water and to establish the optimum curing time and temperature to yield the highest accelerated strengths. Variations due to differences in cements, aggregate gradations, and admixtures were also studied. Field trials were undertaken concurrently with the laboratory program to confirm the relationship established between accelerated and 28-day compressive strengths. As knowledge and experience accumulated, so did interest in the possibilities of using accelerated strength testing as a standard test in its own right rather than for only predicting the 28-day strength for a particular mix on a particular job.

TABLE 2.—PHYSICAL AND CHEMICAL TESTS ON CEMENTS (FIXED SET ACCELERATED CURING PROCEDURE).

CEMENT SOURCE & BATCH Nº	LAB. SAMPLE Nº	PHYSICAL TESTS						CHEMICAL TESTS						
		NORMAL CONSISTENCY %	FINENESS		AUTOCLAVE EXPANSION %	VICAT INITIAL SET FINAL SET hr.-min.	COMP. STRENGTH (psi) 3 DAY 7 DAY 28 DAY	LOSS ON IGNITION %	INSOLUBLE RESIDUE %	TRICALCIUM ALUMINATE %	SULPHUR TRIOXIDE %	MAGNESIA %	SODIUM OXIDE %	POTASSIUM OXIDE %
			RET. #200 %	BLAINE $cm^2/gm.$										
PROVISIONAL ONTARIO ACCELERATED CURING PROCEDURE														
1-3	62-B 56817	23.5	5.2	3131	0.07	2-45 4-15	2417 3233 4842	1.38	0.15	7.76	2.12	2.45	0.53	0.52
x) 1-4	63-B 56808	27.0	1.0	5070	0.04	2-25 3-50	3117 4217 xx)	1.30	0.24	9.37	2.76	1.84	0.54	0.52
2-2	63-B 56807	24.0	7.2	3046	0.18	2-35 3-45	2475 3550 4908	1.23	0.44	10.38	2.46	1.84	0.24	0.97
3-2	63-B 56805	24.5	5.2	3191	0.05	2-15 3-45	2042 2833 4583	0.85	0.28	8.44	2.65	3.35	0.47	0.78
4-2	63-B 56806	24.0	9.2	3183	0.20	2-15 3-45	2617 3542 4675	1.02	0.20	10.97	2.56	1.32	0.55	0.77
6-1	63-B 56804	24.5	8.4	3496	0.03	2-45 4-00	2067 2933 4566	2.32	0.28	9.41	2.36	1.16	0.47	0.67

x) Type III; high early strength cement.
xx) I and 3 day compressive strengths.

MATERIALS USED IN LABORATORY TESTS

Fine Aggregate:

The fine aggregate used in all the tests was a natural sand from Paris, Ont., having a fineness modulus of 2.70, a saturated surface dry bulk specific gravity of 2.70, and an absorption of 1.08 per cent. Where the effect of fineness of the fine aggregate was a variable, it was sieved into six sizes and then recombined to produce the desired fineness modulus.

Coarse Aggregate:

The coarse aggregate was a partly crushed gravel consisting predominantly of hard limestone from Brighton, Ont. Its saturated surface dry bulk specific gravity was 2.70, absorption 0.40 per cent, and dry rodded unit weight 110 lb per cu ft.

Except where the variable was the nominal maximum size, the coarse aggregate was separated into four fractions and then recombined to produce the following grading:

Tyler Standard Sieve Size	Cumulative Per Cent Passing
1.050 in.	100
0.742 in.	90
0.525 in.	60
0.371 in.	30
4 mesh	0

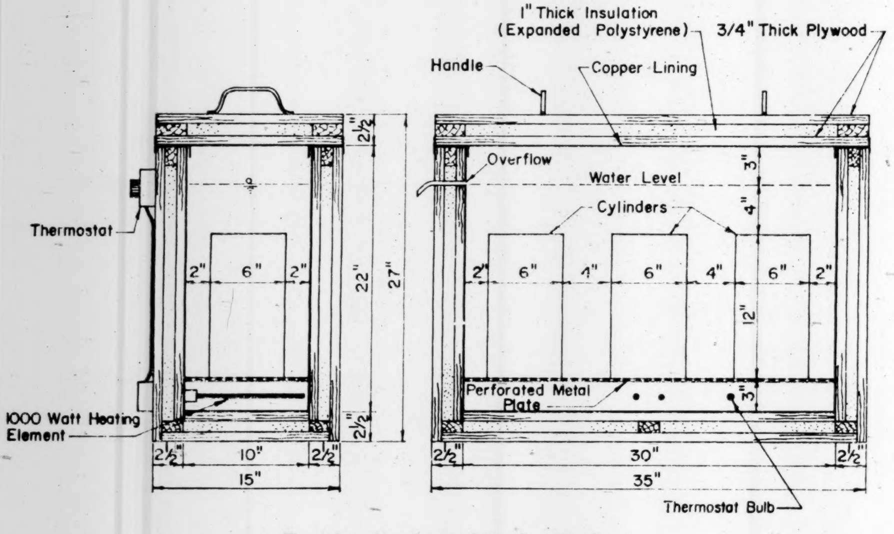
1" Thick Insulation (Expanded Polystyrene)
3/4" Thick Plywood
Handle
Copper Lining
Overflow
Water Level
Cylinders
Thermostat
1000 Watt Heating Element
Perforated Metal Plate
Thermostat Bulb
2½"
22"
27"
2"
6"
2"
2½"
10"
2½"
15"
3"
4"
2"
6"
4"
6"
4"
6"
2"
12"
3"
2½"
30"
2½"
35"

FIG. 1.—Accelerated Curing Tank.

Cements:

Cements from six Ontario mills were used in the tests. Standard chemical and physical tests were performed on each cement; the results obtained are given in Tables 1 and 2. Each cement is identified by two figures, for example, cement 1-2; the first figure identifies the source, the second the batch number. All were type I cements with the exception of cement 1-4 which was type III.

The time of setting was measured with the Vicat apparatus. The initial set was determined in accordance with ASTM Method C 191;[3] the final set was obtained when the needle did not sink visibly into the paste.

Admixtures:

Twelve admixtures were used in the tests. Admixtures A, B, C, D, E, F, G, H, and I were of the lignosulfonate type, admixture J was of the hydroxy-carboxylic acid type, K was a neutralized vinsol resin air-entraining agent, and L was calcium chloride.

Admixtures A to H were water-reducers claimed not to cause a significant change in setting time of the concrete while admixtures I and J were intended to produce retardation.

EQUIPMENT AND GENERAL PROCEDURE USED

All concrete was mixed and tested by standard equipment and methods except, as will be described later, a bond pin pullout apparatus was used to measure the set of the concrete in certain tests.

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[3] Method of Test for Time of Setting of Hydraulic Cement by Vicat Needle (C 191), 1961 Book of ASTM Standards, Part 4.

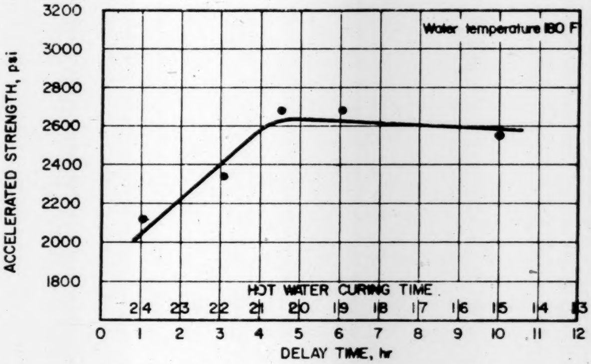


FIG. 2.—Exploratory Tests. Delay Time and Hot-Water Curing Time versus Accelerated Strength

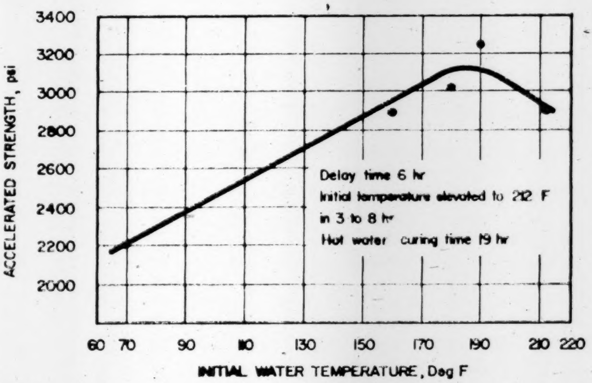


FIG. 3.—Exploratory Tests. Initial Water Temperature versus Accelerated Strength.

A moist room at 72 F ± 2 F and approximately 100 per cent relative humidity was used for normal curing. For accelerated curing, a water tank of the design and construction shown in Fig. 1 was used, the temperature of the water was thermostatically controlled, heating was by a 1000-w electrical immersion heater. Four such tanks were used, each holding three cylinders. During the early stage of the investigation a tank of larger inside dimensions (30 by 16 by 24 in.) was used.

In the first stage of the laboratory investigation, cast iron cylinder molds were used while in the second stage and in the field tests, metal molds were used. During the delay time (time elapsed from mixing the concrete to placing the cylinders in the water tank) the molds were covered with steel plates and wet burlap. The concrete remained in the molds, still covered with the steel plate throughout the hot water curing time (time elapsed from placing the cylinders in the tank until their removal).

Upon removal of the cylinders from the tank, the molds were at once stripped. The cylinders were then allowed to stand at room temperature (72 F) for half an hour to cool before capping with a sulfur-granular material. They were then tested in compression 1 hr after removal from the tank.

In the laboratory tests, normally cured cylinders were kept in their molds for 24 ± 2 hr and for 27 days without their molds in the moist room. In the field, such cylinders were kept in their molds for 24 ± 2 hr under wet burlap and for 27 days without their molds in a tank of lime-saturated water at approximately 70 F.

All tests were based on at least three cylinders for each variable examined. All cylinders, both those whose curing was accelerated and those cured normally were made from the same batch of concrete for each variable tested.

STANDARD-DELAY ACCELERATED CURING PROCEDURE (SDAC PROCEDURE)

To establish an accelerated curing procedure, the first series of exploratory tests investigated the effect of delay time, hot water curing time, and initial water temperature. Non-air-entrained concrete without any admixtures was used. In all mixes, the cement content was approximately 525 lb per cu yd (cement 1:1), the water cement ratio 0.50 by weight with a sand-aggregate ratio of 0.42 by solid volume.

The results of the exploratory tests are shown in Figs. 2 and 3. Based on these, the following accelerated curing procedure, which appeared to offer convenient timing and a high level of strength development, was established.

1. Cover the cast iron molds with steel plates immediately after making the cylinders.
2. Place the cylinders under wet burlap for 6 hr.
3. Place the cylinders, complete with molds and steel plates, in water at 190 F and bring to the boil in $5\frac{1}{2}$ to $6\frac{1}{2}$ hr.
4. After 19 hr in hot water, remove the cylinders from the tank, strip the molds, allow to cool for approximately half an hour, then cap with a sulfur-granular material.
5. Test in compression 1 hr after removal from the tank.

The total time for this procedure is 26 hr.

This procedure was then used to determine the relationship of accelerated to 28-day strengths and the effect of concrete variables on this relationship. Among the variables examined were water-cement ratio, cement content, air content, maximum size of coarse aggregate, and fineness modulus of the sand. As will be shown, these did not appear to affect the relationship of accelerated to 28-day strength either in the laboratory or in the field trials. Two other variables did, however, have a definite and disturbing effect on this relationship. The effects of different sources and batches of cements and of various water-reducing and retarding admixtures are therefore reported separately.

RELATIONSHIP BETWEEN ACCELERATED AND 28-DAY STRENGTHS USING SDAC PROCEDURE FOR NON-RETARDED CONCRETE WITH THE SAME CEMENT (1–1)

The following mixes were made:

1. Twenty-one mixes with different cement contents and water-cement ratios, air content 6.0 ± 1.0 per cent (admixture K), sand-aggregate ratio by solid volume 0.42:

Cement Content, lb per cu yd of concrete	Aggregate-Cement Ratio by Solid Volume	Water-Cement Ratio by Weight
350	11.1	0.52; 0.56; 0.61; 0.66; 0.69
437.5	8.7	0.55; 0.60; 0.65; 0.70
525	7.1	0.45; 0.50; 0.55; 0.60
612.5	5.9	0.40; 0.45; 0.50; 0.55
700	5.0	0.35; 0.40; 0.45; 0.50

2. Six mixes with different air contents (2.6, 3.7, 5.7, 8.1, 9.8, and 11.3 per cent), using a cement content of approximately 525 lb per cu yd of concrete, constant sand-aggregate ratio, constant water-cement ratio 0.45, and admixture K as an air-entraining agent.

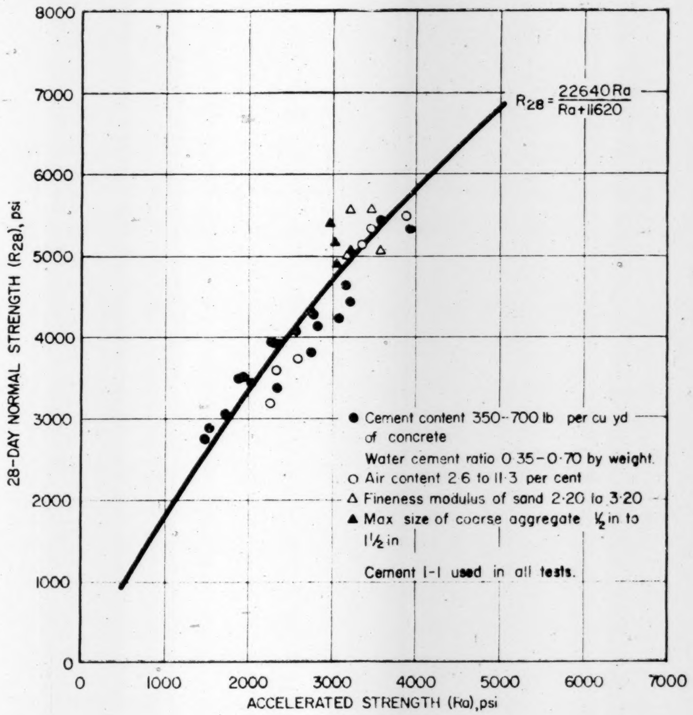


FIG. 4.—Standard Delay Accelerated Curing Procedure. Relationship between Accelerated and 28-Day Normal Strengths.

3. Four non-air-entrained mixes with different maximum sizes (1½, ¾, and ½ in.) of coarse aggregate using a cement content of approximately 525 lb per cu yd of concrete and constant slump.

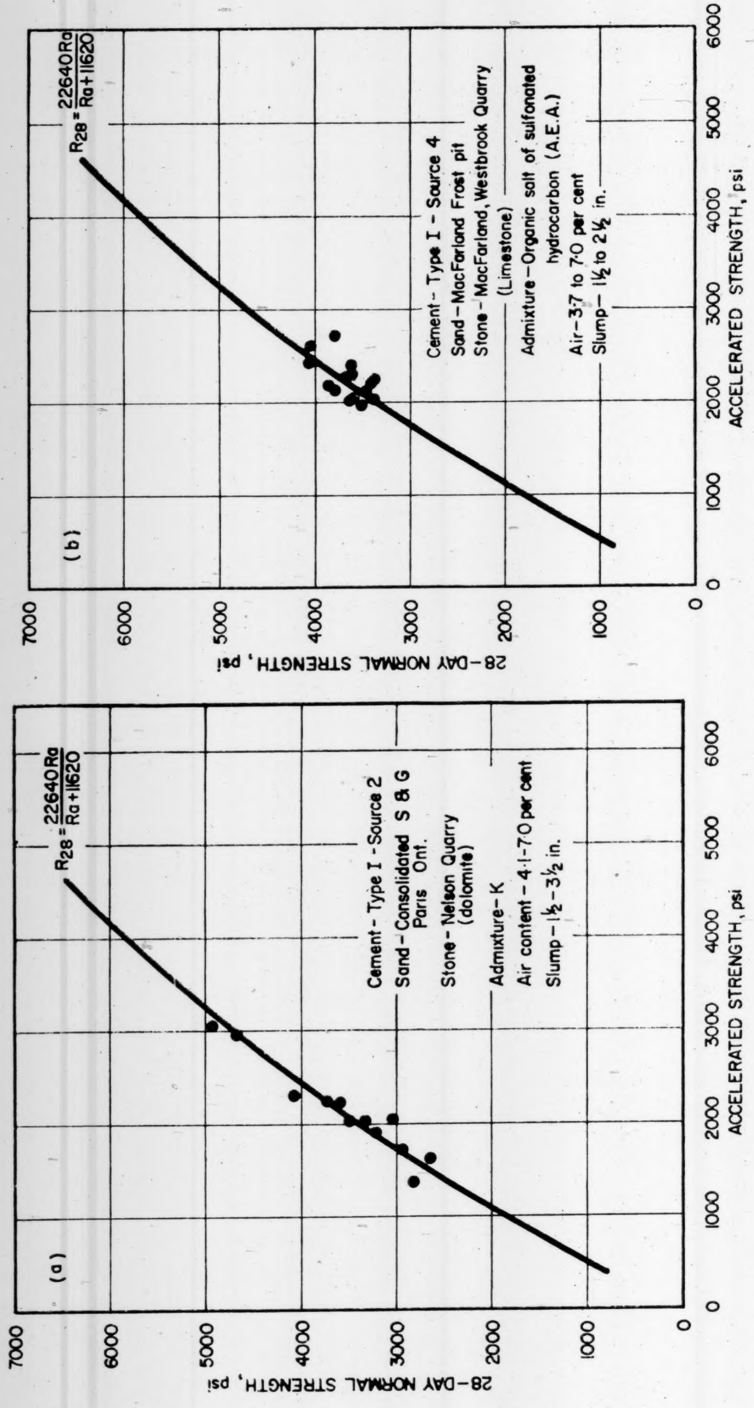
4. Four non-air-entrained mixes with different fineness moduli (2.20, 2.55, 2.90 and 3.20) of fine aggregate, using a cement content of approximately 525 lb per cu yd of concrete and constant slump.

The results of these tests are shown in Fig 4. If the relationship is assumed to be hyperbolic, it can be expressed by the following equation derived by the method of least squares, the standard deviation being 333 psi:

$$R_{28} = \frac{22640\ R_a}{R_a + 11620}$$

where R_{28} is the 28-day strength and R_a is the accelerated strength.

Possibly too few mixes were made with varying air content, maximum size of coarse aggregate and fineness modulus of the sand, to establish with certainty whether these had any effect on the relationship. All points were, however, close to the regression curve, and there is no reason to suspect that these variables would have any effect.



(a) Field Trial 1.

(b) Field Trial 2.

FIG. 5.—Standard Delay Accelerated Curing Procedure.

Field Trials:

To see if the relationship established in the laboratory still held under practical conditions, field trials using the same SDAC Procedure were arranged on a number of concrete paving contracts.

The accelerated curing tanks were set up in field laboratories which were 10 to 15 miles from the job site. The cylinders for both accelerated and normal curing were transported to the laboratory during the early part of the delay time with no apparent harm.

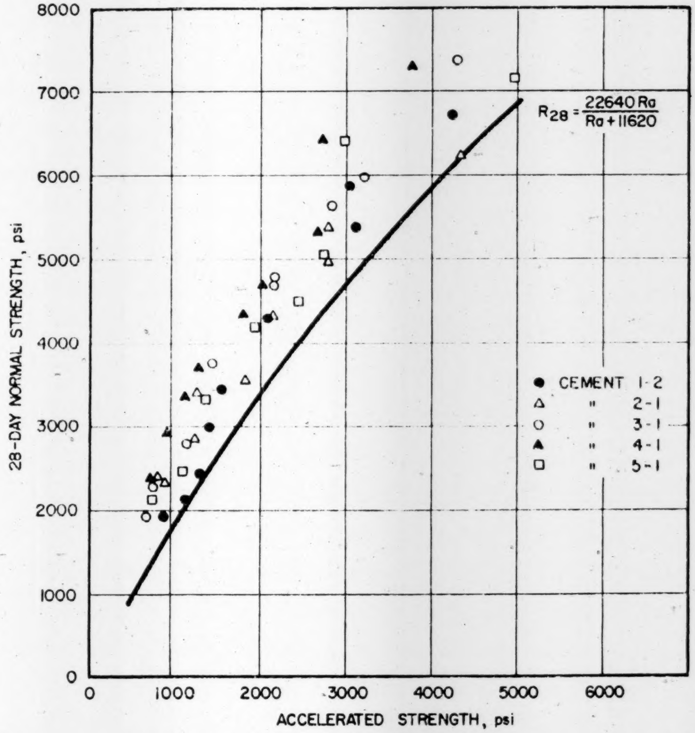


FIG. 6.—Standard Delay Accelerated Curing Procedure. Effect of Cements from Different Sources.

The concretes tested contained approximately 570 lb of cement per cu yd and were air entrained with either a neutralized Vinsol resin or organic salt of sulfonated hydrocarbon type air entraining agent. Normal job variations in mix proportions, aggregate gradings air contents, slumps, etc., occurred during the work.

As Figs. 5(*a*) and (*b*) show, the accelerated to 28-day strength points are all very close to the regression line established in the laboratory.

Effect of Cements from Different Sources:

Nine non-air-entrained concrete mixes were made with each of five different cements (1-2, 2-1, 3-1, 4-1, 5-1) as follows:

Cement Content, lb per cu yd of concrete	Water-Cement Ratio by Weight
350..........	0.60; 0.70; 0.80
525..........	0.45; 0.55; 0.65
700..........	0.35; 0.45; 0.55

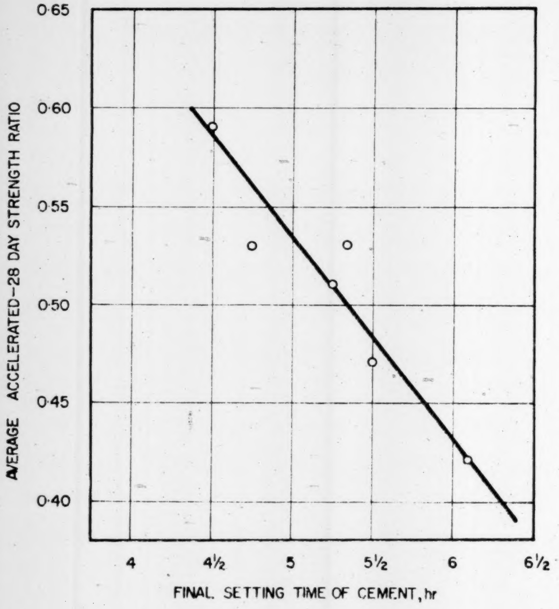


FIG. 7.—Standard Delay Accelerated Curing Procedure. Final Setting Time of Cement versus Average Accelerated and 28-Day Strength Ratio.

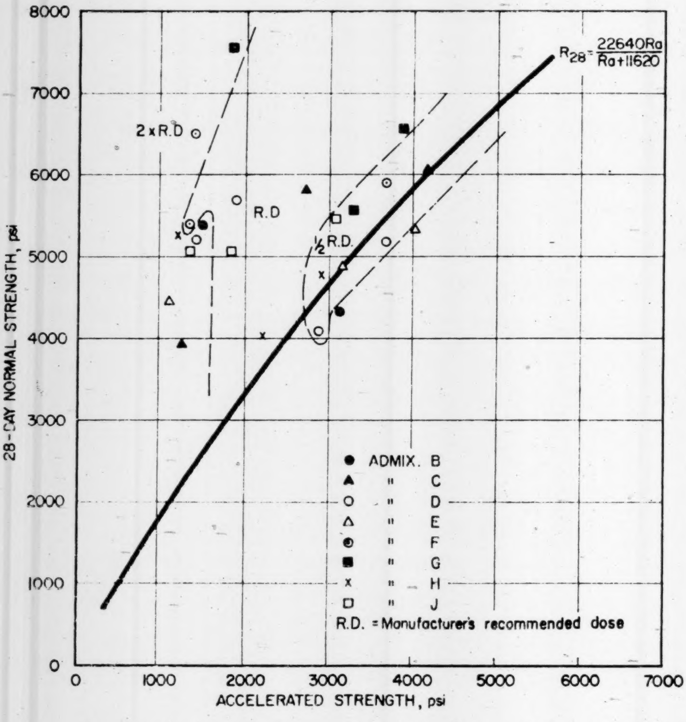


FIG. 8.—Standard Delay Accelerated Curing Procedure. Effect of Admixtures.

The results of these tests are plotted in Fig. 6. Almost all the points fall above the regression curve established using cement 1-1, and each cement tends to have its own accelerated to 28-day strength relationship. This effect appears to be related to the setting characteristics of the cements; the cements with long final setting times having a lower average accelerated to 28-day strength ratio (Fig. 7).

Effect of Water Reducing and Retarding Admixtures:

Each of admixtures B, C, D, E, F, G, H, and J were incorporated separately at half, once, and twice the manufacturers' recommended dose in concrete mixes containing approximately 525 lb cement per cu yd, the slump was held at 3 ± $\frac{1}{2}$ in. with the air content varying from 3.4 to 11.4 per cent.

From Fig. 8, it can be seen that with half the recommended dose of admixture, the accelerated to 28-day strength plots are reasonably close to the regression line for the general relationship previously established. However, with the recommended dose of most of the admixtures and with twice the recommended dose of all admixtures the points are far above the regression line. Examination of such cylinders showed expansion of the concrete, in some cases of as much as $\frac{1}{2}$ in. The indication was that the cylinders had been placed in the hot water for accelerated curing before they had set sufficiently.

RELATIONSHIP BETWEEN DEGREE OF SET AT THE START OF HOT WATER CURING, WATER TEMPERATURE, AND ACCELERATED STRENGTH

The tests performed on concretes made with different cements and admixtures using the SDAC Procedure with its short standard delay time suggested that a universally applicable relationship between accelerated and 28-day strengths was not attainable. Further tests were therefore undertaken to clarify the effects of delay time, degree of set, and water temperature on the relationship.

Effect of Extending the Delay Time:

In this series of tests, a non-air-entrained concrete containing approximately 525 lb of cement 1-3 per cu yd with a water-cement ratio of 0.50 by weight, and sand-aggregate ratio of 0.42 by solid volume was used.

When tested by the SDAC Procedure, this concrete produced an average accelerated strength of 2800 psi. However, when the delay time was increased to 21 hr and the hot-water curing time was reduced to 6 hr, the accelerated strength was only 1700 psi. Almost the same strength, 1650 psi, was achieved by 28 hr of normal moist curing.

With a higher initial water temperature of 212 F, a delay time of 18, 21, or 24 hr and a hot-water curing time of 24 hr, the accelerated strength was in each case 2530 psi. After 48 hr of normal moist curing, the strength was 2540 psi.

It was therefore apparent that arbitrarily extending the delay time to a positively safe point still convenient to the working day negated any acceleration in strength development.

Effect of Degree of Set and Initial Water Temperature:

When the tests described in this paper were started, the laboratory was not equipped to carry out ASTM Method C 403, Method of Test for Time of Setting of Concrete Mixtures by Penetration Resistance.[4] A pin pull-out apparatus (6) was available, and this was used to determine the time of set in the majority of the tests.

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[4] 1962 Supplement to Book of ASTM Standards, Part 4.

From each batch of concrete, a 6 by 6 by 36-in. specimen with 17 stainless steel pins embedded from $5\frac{1}{2}$ to $5\frac{3}{4}$ in. in the concrete was made. The $\frac{3}{8}$-in. diameter pins were pulled out of the concrete at different time intervals depending on the rate at which the bond strength increased. At the beginning of the test, one pin was pulled out each hour, at the end of the test every 5 min. The bond strength was plotted against the time elapsed since mixing the concrete. From this graph, the bond strength at the time of placing the cylinders in the hot water tank was interpolated.

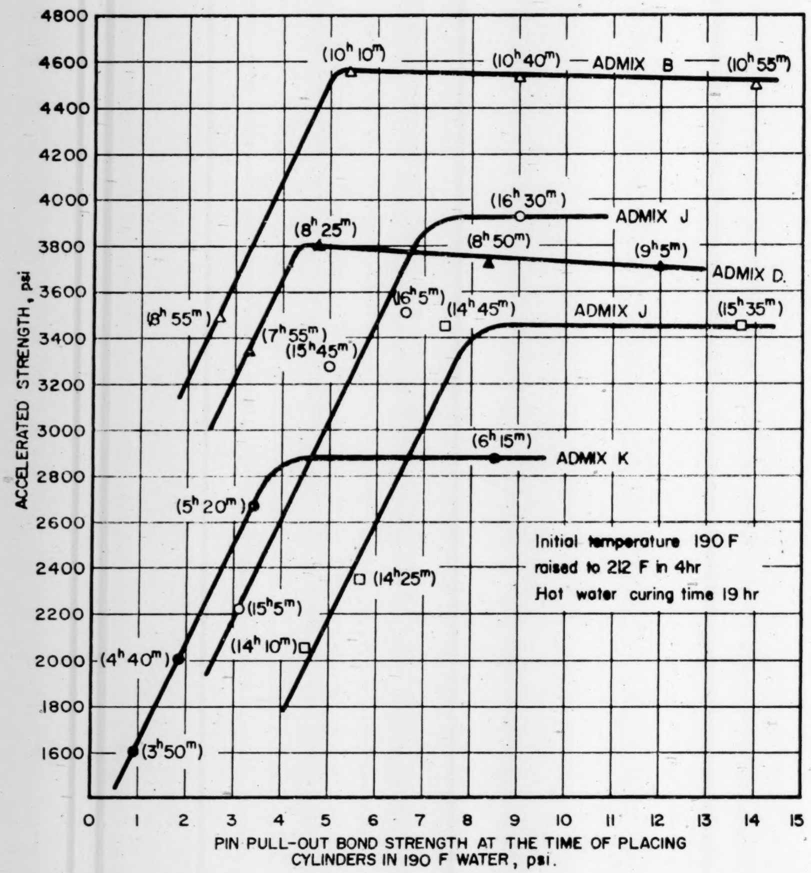

FIG. 9.—Pin Pull-Out Bond Strengta of the Concrete at the Time of Placing Cylinders in 190 F Water versus Accelerated Strength.

Five mixes were made, one mix contained admixture B, one contained admixture D, and two contained admixture J. The fifth mix did not contain any water-reducing admixture. It was, however, likewise air entrained with admixture K. The cement content of each mix was approximately 525 lb per cu yd of concrete, air content was 6 ± 1 per cent, and the slump was 2 to 4 in. From each mix, 12 cylinders and one pin pull-out specimen were made.

The cylinders were placed in 190 F water when the concrete had reached the pin pull-out bond strengths shown in Fig.

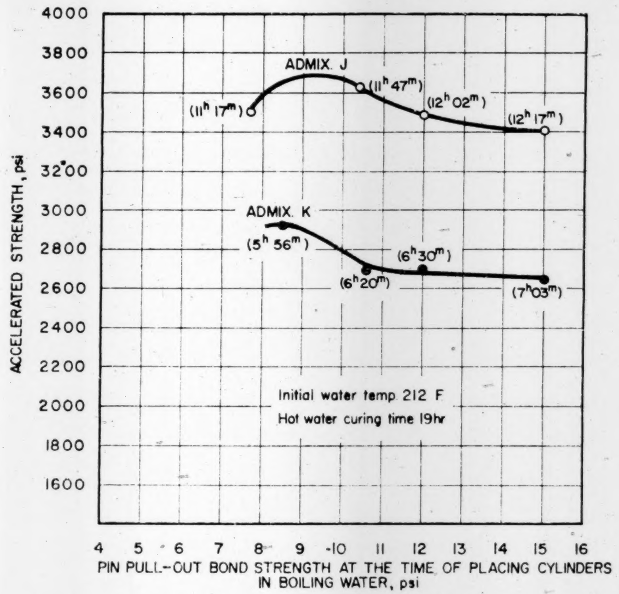

FIG. 10.—Pin Pull-Out Bond Strength of the Concrete at the Time of Placing Cylinders in Boiling Water versus Accelerated Strength.

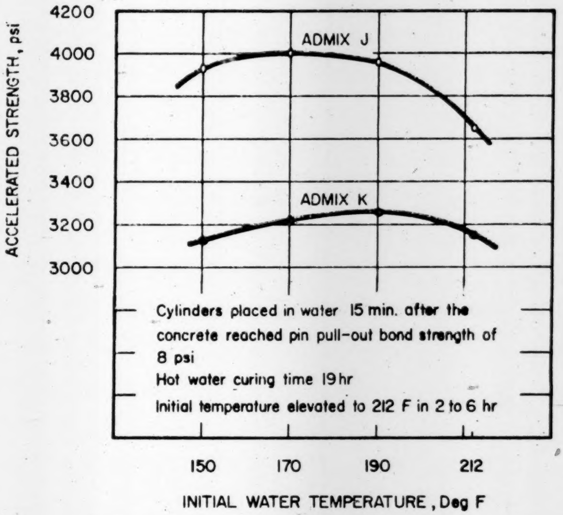

FIG. 11.—Initial Water Temperature versus Accelerated Strength.

9. The delay time therefore varied, otherwise the SDAC procedure was followed.

The results of these tests (Fig. 9) showed that the accelerated strength reached a maximum value which depended on the degree of set of the concrete at the time of placing cylinders in the hot water and that the pin pull-out bond strength at which the curves levelled off occurred between 4 and 8 psi, depending on the admixture used.

If instead of an initial temperature of

190 F, the water was boiling when the cylinders were placed in the tank the relationship between the pin pull-out bond strength and accelerated strength was as shown in Fig. 10. The curves in this case level off at a bond strength of approximately 12 psi.

To study the effect of initial water temperature further, mixes containing admixture J (retarded) and admixture K (non-retarded air-entrained) were made. Sets of cylinders were placed in water tanks at 150 F, 170 F, 190 F, and 212 F at 15 min after the concrete had reached a pin pull-out strength of 8 psi; otherwise the SDAC Procedure was used, the water being brought to the boil in from 2 to 6 hr. The effect of the rate of temperature rise was not investigated; while the effect is likely to be small it should be checked.

As Fig. 11 shows, for both retarded and non-retarded air-entrained concrete the 150 F, 170 F, and 190 F initial temperatures had little effect on the accelerated strengths, while 212 F gave a lower accelerated strength with the retarded concrete. This indicated that such concrete had probably not reached a sufficient degree of set when placed in the boiling water and confirmed the wisdom of selecting a pin pull-out bond strength of 12 psi + 20 min as the measure of delay before cylinders might safely be placed in boiling water.

The reason for preferring boiling water over water at an initial temperature of 190 F subsequently brought to the boil is that only one large accelerated curing tank kept at constant boil is required. Additional cylinders can then be placed in the tank as and when required.

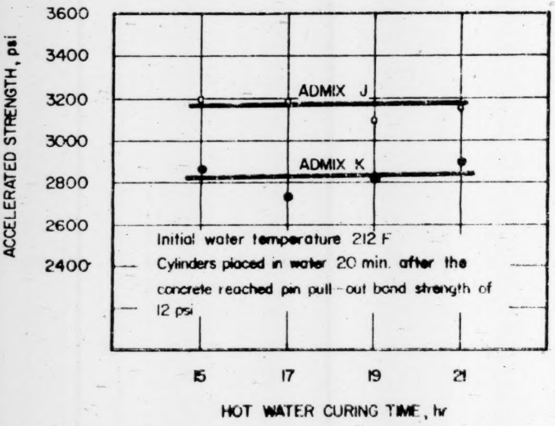

FIG. 12.—Hot-Water Curing Time versus Accelerated Strength.

Effect of Hot-Water Curing Time:

Two mixes were made similar to those described in the preceding section. The initial water temperature was 212 F, the cylinders were placed in the water 20 min after the concrete had reached a pin pull-out bond strength of 12 psi.

As the results in Fig. 12 show, there is a negligible difference in the accelerated strength of cylinders cured in boiling water for from 15 to 21 hr. Most concretes without retarders attain a pin pull-out bond strength of 12 psi in 6 to 9 hr after mixing the concrete; therefore a 16-hr hot-water curing time was selected so that in most cases results can be available approximately 24 hr after mixing the concrete.

FIXED-SET ACCELERATED CURING PROCEDURE (FSAC PROCEDURE)

The above tests established an accelerated curing procedure in which the delay time was based on the concrete having attained a fixed degree of set. The following procedure has thereafter proved suitable for all concretes tested to date regardless of their proportions or the effect of different cements and admixtures:

1. Cover the metal molds with steel plates immediately after making the cylinders and place the cylinders under wet burlap.
2. Place the cylinders complete with molds and steel plates in boiling water 20 min after the concrete reaches a needle penetration resistance of 3500 psi or pin pull-out bond strength of 12 psi.
3. Maintain the cylinders in the water tank for 16 hr.
4. Remove the cylinders from the tank, strip the molds, allow to cool for approximately half an hour and cap with a sulfur-granular material.
5. Test in compression one hour after removal from the boiling water.

At the time of making cylinders, either:

(*a*) A pin pull-out specimen is made from the same batch of concrete as the cylinders and the pin pull-out bond strength tested; or

(*b*) The Proctor needle penetration resistance is determined in accordance with ASTM Method C 403. A Proctor needle penetration resistance of approximately 3500 psi is equivalent to a pin pull-out bond strength of 12 psi.

After placing the cylinders in the boiling water, the water should be returned to the boil in 2 hr ± 5 min. This can be accomplished by using well-insulated tanks (Fig. 1) and maintaining a constant volume of water in the tanks.

This procedure was then used to determine the relationship of accelerated to 28-day strengths and the effect of such variables as different cement contents, water cement ratios, air contents, and water reducing and retarding admixtures.

RELATIONSHIP OF ACCELERATED TO 28-DAY STRENGTHS USING FSAC PROCEDURE FOR RETARDED AND NON-RETARDED CONCRETES MADE WITH THE SAME CEMENT 1–3

The following mixes were made:

1. Twenty mixes with different cement contents and water-cement ratios using non-air-entrained concrete with constant coarse aggregate content; sand content varied according to the cement and water contents:

Cement Content, lb per cu yd of concrete	Water-Cement Ratio by Weight
350	0.65; 0.70; 0.80; 0.90
437.5	0.55; 0.60; 0.65; 0.70
525	0.45; 0.50; 0.55; 0.60
612.5	0.40; 0.45; 0.50; 0.55
700	0.35; 0.40; 0.45; 0.50

2. Ten mixes with different air contents (air entraining admixture K) and sand-aggregate ratios, slump 3 ± ½ in. and constant cement content of approximately 525 lb per cu yd of concrete:

Air Content, per cent	Sand-Aggregate Ratio by Solid Volume
3.8; 4.4	0.43
4.8; 6.1	0.42
6.7; 6.8	0.41
7.4; 9.3	0.40
10.0; 10.1	0.39

3. Eighteen mixes with admixtures A, C, D, H, I, J, and L, cement content was approximately 525 lb per cu yd of concrete, slump from 2⅞ to 4½ in., air content from 5.0 to 9.7 per cent depending on the admixture and the dose used. The water-cement ratio by weight was within 0.37 to 0.57. Three mixes were made with each admixture using ½, 1,

and twice the manufacturers' recommended dose. For admixture I only two and for admixture L only one mix was made.

A hyperbolic curve was fitted to the accelerated and 28-day strength points obtained (Fig. 13), using the least squares method, the standard deviation being 347 psi.

The equation $R_{28} = (26160\,R_a)/(R_a + 11620)$ represents the relationship. The straight line $R_{28} = 1.507\,R_a + 820$ does not fit the points as well as the hyperbolic curve, but it can be used for the calculation of R_{28} when R_a is between 1500 and 3500 psi.

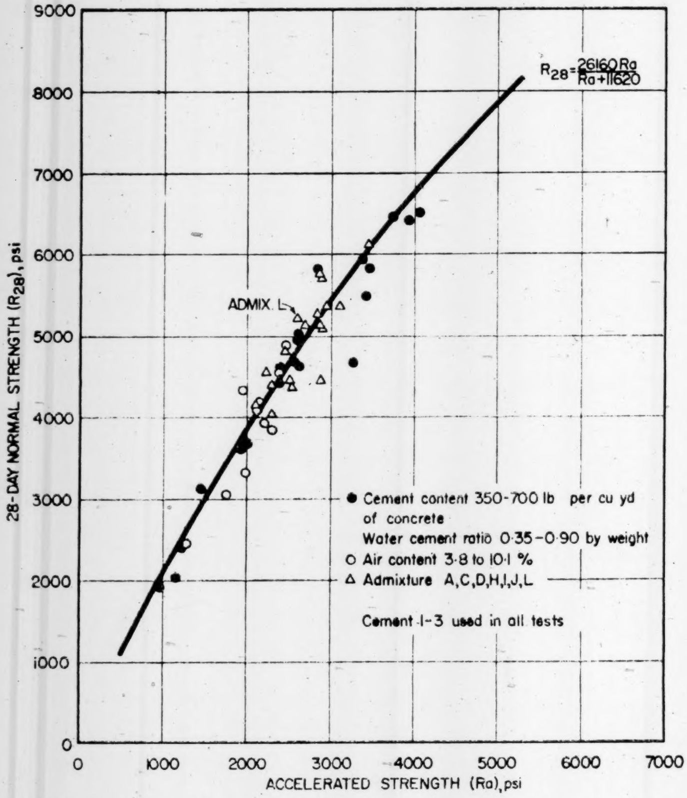

FIG. 13.—Fixed-Set Accelerated Curing Procedure. Relationship between Accelerated and 28-Day Normal Strengths.

Effect of Different Cements:

The effect of different cements on the accelerated to 28-day strength relationship was tested using five cements of which one (1-4) was a type III cement and four (2-2, 3-2, 4-2, 6-1) were type I cements. Six air-entrained mixes were made with each cement. The cement content and water-cement ratio were varied as follows:

Cement Content, lb per cu yd of concrete	Water-Cement Ratio by Weight
350...........	0.70; 0.80
525...........	0.50; 0.60
700...........	0.35; 0.40

Two non-air-entrained mixes were made with each of cements 2-2, 3-2, and 6-1. The cement content was approximately 700 lb per cu yd of concrete with water-cement ratios of 0.35 and 0.40 by weight. One non-air-entrained mix was made with cement 4-2. It had a cement content of approximately 700 lb per cu yd of concrete and a water-cement ratio by weight 0.35.

In all these mixes the coarse aggregate content was constant; sand content varied according to the cement and water contents. Air contents were between 2.0 and 7.4 per cent.

The test results are shown in Fig. 14. The accelerated to 28-day strength points for the concretes with the different brands of cement including the type III fit the regression line established for the concretes containing cement 1-3.

Field Trial of FSAC Procedure:

During the summer of 1963, a field trial covering approximately 10,000 cu yd of concrete was arranged on a bridge deck contract. Sets of cylinders for accelerated and normal curing were made representative of each separate concrete placement. The concrete samples were transported to an adjacent field laboratory where the cylinders were made, cured, and tested. The degree of set of the concrete was measured in accordance with ASTM Method C 403. The 3500 psi Proctor needle penetration resistance was reached in from 6 to 12 hr after mixing, depending

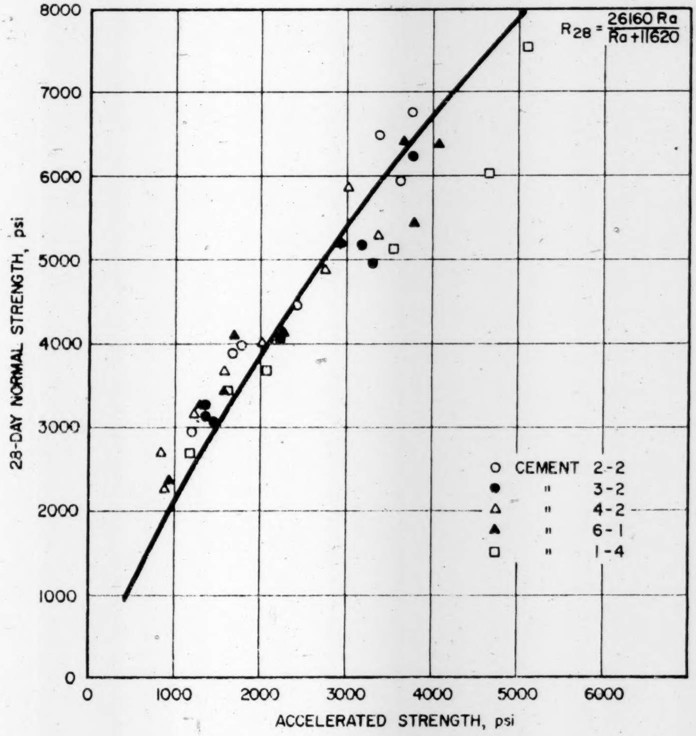

FIG. 14.—Fixed-Set Accelerated Curing Procedure. Tests on Different Brands of Cement.

on the temperature, the admixture, and the dose used.

All the concrete contained approximately 525 lb cement per cu yd. The cement was all type I from source 3 except for 2 placements which contained a type I cement from source 2. Fourteen concrete placements contained an air-entraining agent only. Twenty contained a water-reducing retarding admixture and air-entraining agent, and the two which used cement from source 2 contained a water-reducing non-retarding admixture and air-entraining agent. Both admixtures were of the calcium lignosulfonate type, and the air-entraining agent was an organic salt of a sulfonated hydrocarbon. On many of the hotter days, ice was used with the mixing water to keep concrete placement temperatures below 80 F. Two coarse aggregates were used; both dolomitic limestones, and the sand was the same throughout.

The results are shown in Fig. 15. Most accelerated to 28-day strength points are close to the regression line established using the FSAC procedure in the laboratory.

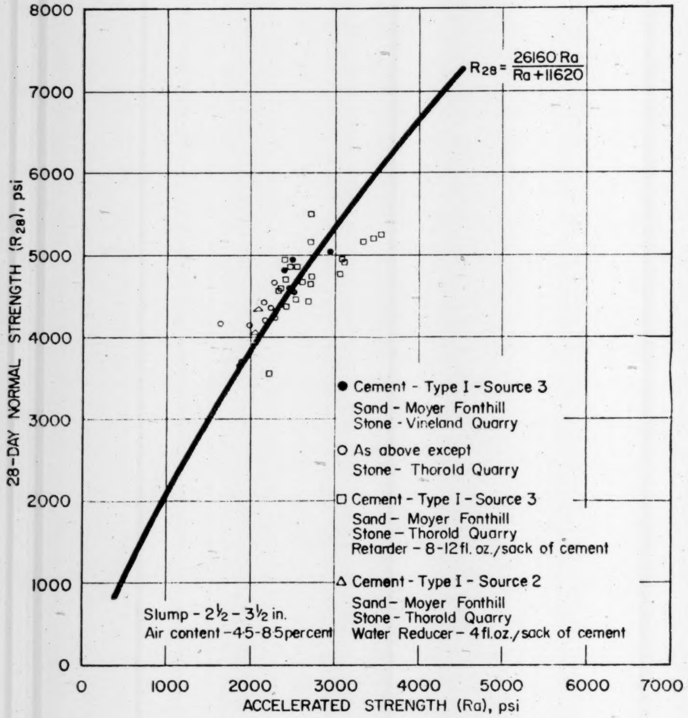

FIG. 15.—Fixed-Set Accelerated Curing Procedure. Field Trial.

GENERAL DISCUSSION

The laboratory tests and field trials show that FSAC Procedure is capable of yielding high accelerated strengths while taking into account such variations as arise from the setting characteristics of the concrete. Results appear to be reproducible, and the equipment required is simple and usable both in the laboratory and field. The big disadvantage seems to be in that extra work is required to determine the setting time of the concrete; this and placing the cylinders in the water tank probably involves overtime work when the concrete is mixed after mid-morning.

At first sight, this makes the alternative of normal curing the cylinders until next day before starting acceleration attractive. This is basically the modified procedure developed by Akroyd (7) in which cubes after 24-hr standard curing are boiled for 3½ hr. Unfortunately, as has been shown, with such an extended delay normal curing alone for the same total time would give almost the same strength development and this would be much lower than that achieved by the FSAC Procedure. The other alternative of lowering the initial water temperature so that cylinders might be placed in the tank before they are set also leads to lower accelerated strengths and more difficult control of the accelerating procedure.

Capping Cylinders:

The accelerated to 28-day strength plots in Figs. 4, 6, 14, and 15 show greater scatter above an accelerated strength of approximately 3000 psi than below. This may be due to one or more reasons:

1. Dependence of standard deviation on strength level.
2. Caps on 28-day cylinders even after 16-hr hardening are weaker than the cylinders themselves in the above 3000-psi range.
3. The caps on the accelerated cylinders were cast only ½ hr after their removal from the tank and were only ½ hr old when the cylinders were tested.
4. An effect of the aggregate.

It is unlikely, since the coarse aggregate was of good quality, that this was the cause of the greatest scatter. Therefore an investigation should be undertaken into capping materials and methods as related to the special needs of accelerated strength testing or into the use of horizontal cylinder molds. In this respect, the British are fortunate in their use of cubes which only need flipping onto their side for testing. For concretes in the medium and lower strength range, cylinder capping would not appear to be a problem.

Variations Due to Aggregates:

While the FSAC Procedure takes account of variations that affect the setting time of the concrete, it cannot, nor can any accelerated procedure which does not develop the full 28-day strength potential of the paste, detect such effects as the presence of low-strength aggregates. A specific laboratory investigation into effects due to aggregates has not yet been made. However, gravel, crushed limestone, and dolomite aggregates have been used in the concretes tested, and no effect on the relationship of accelerated to 28-day strengths due to these differences or differences in maximum size or grading has been observed. Akroyd (7), on the other hand, showed that while different aggregates do affect the results, an accelerated to 28-day strength relationship still exists.

The effect of aggregates should be further investigated. Aggregates which are of poor strength are probably well known in a particular locality and are customarily not used in high-strength mixes, or could be detected if trial mixes are made prior to starting the job.

Practical Use of Accelerated Strength Testing:

Being able to estimate the 28-day strength of concrete after approximately 24 hr is in itself valuable. Yet the results may be even more useful if they will permit the operation of an over-all quality control system based on their evaluation in the manner recommended by ACI Committee 214 (8). The implications of using accelerated strengths for this purpose should be investigated to see if at least an equal degree of confidence would apply.

It should be pointed out that the use of accelerated strength for quality control purposes is self checking. If a large enough volume of concrete is involved over a long enough time, a correlation history is established as soon as sufficient 28-day strengths are available. Otherwise if prior proof of such correlation is needed—for example, because the aggregates are suspected to be below par—then extra cylinders can be made for accelerated curing at the mix design stage.

When the mix is first used on the job, if the accelerated strengths of field cylinders differ significantly from the value taken from the standard relationship or the datum figure established on the trial mix, it is known next day that the design 28-day strength will be met, exceeded, or not realized. In the last two eventualities, this can be an extremely valuable warning which allows immediate corrective steps to be taken.

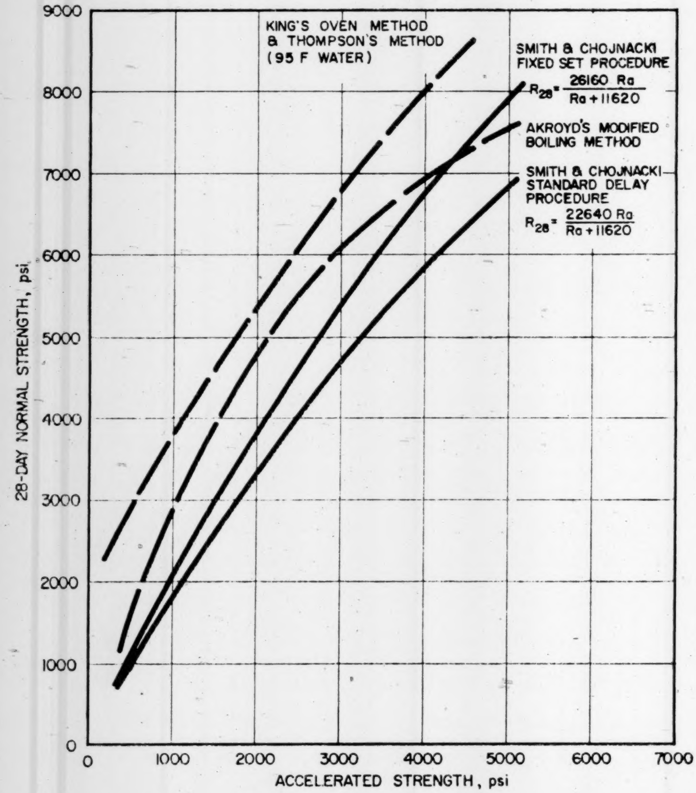

FIG. 16.—Comparisons of Accelerated Curing Methods.

COMPARISON BETWEEN THE PROCEDURE DEVELOPED AND THOSE USED BY OTHERS

Figure 16 draws a comparison between the SDAC and FSAC Procedures and three of the procedures having greatest advocacy in England.

Each of these English procedures has involved variations in the accelerating medium. King and co-workers (**3,4,9–11**) have advocated heating the specimens while covered and still within their molds by dry heat in an oven. Usually the concrete cubes have been placed in a cool oven half an hour after mixing. The oven is then brought to 200 F within 2 hr and so maintained to give a total heating time of 6 hr. The cubes are then tested half an hour after removal from the oven. This gives a testing time of 7 hr. Modifications of this basic procedure have offered a range of cycles more convenient to fit the working day. The original work of Akroyd and Smith-Gander (**5**) was based on $8\frac{1}{2}$ hr total testing time. Half an hour after mixing, the cubes were placed in water at 140 F which was then brought to the boil. After 7 hr in the water, the cubes were removed, cooled, and tested 1 hr later. Akroyd's later work (**7**) modified this cycle to the more convenient one of 24 hr standard curing followed by $3\frac{1}{2}$ hr boiling and testing 1 hr later. Thompson (**12**) settled on the procedure of using hot water curing at 95 F for 24 hr on the basis that this produced accelerated strengths of the same order as King achieved with his oven.

Discussion of these procedures has brought out arguments for and against each. Akroyd (**7,12**) has postulated that lack of uniformity in temperature within an oven aggravated by the variations in the number of specimens means that each operator with each type of oven gets different results. King (**12**), when discussing Akroyd's work, stated that the scatter of results is wider than those he himself obtained, and that the relationship claimed by Akroyd of accelerated to 7-day = 7-day to 28-day strengths is not borne out by his own and other work. Minor variations in procedures and use have been made by others (**12**) to suit their own needs.

These comparisons appear to show that the procedures developed in Ontario achieve their object of giving the highest possible ratio of accelerated to 28-day strength modified only to the extent necessary to take into account variations in setting time of the concrete.

A Standard Accelerated Strength Test:

The need in England for a standard accelerated curing procedure is evident from the divergent discussion of Akroyd's (**12**) work. A subcommittee of the Institution of Civil Engineers is actively looking into the matter. If later chaos and variation for the sake of variation is to be avoided, similar action in North America would appear wise. Standardization might well involve a different system of acceleration to that advocated in this paper. For example, steam curing might be worth considering. This is already widely used in the prestressed concrete industry, where cylinders steam cured alongside the beams are used to control stress transfer and thus in effect to "accept" the quality of the concrete. This fact led the authors (**12**) when discussing Akroyd's work to suggest the possibilities of using accelerated strength results as an acceptance test for concrete without any reference to the corresponding 28-day strengths.

Compressive strength at 28 days does not measure a property of the final concrete, and where used to specify or judge the quality and strength of concrete in construction it is still only a measure of the potential of the concrete. Such concrete will often not receive adequate, let alone similar, curing to the test cylinders; therefore the value of waiting 28 days to judge its strength is hard to justify if this could be done sooner. In the quality testing of materials, test procedures have been established to measure as simply and rapidly as possible those properties related to use and performance about which information is needed. Examples in the concrete field of such accelerated tests are freezing-and-thawing durability, soundness, and abrasion resistance, for each of which there is an ASTM method of test. The work reported in this paper strengthens the authors' opinion that an accelerated strength test for concrete is a practical possibility.

CONCLUSIONS

It is hoped that this paper will arouse interest in the possibilities of accelerated strength testing.

1. It has been confirmed that it is possible to develop an accelerated curing procedure using boiling water which will allow the compressive strength of concrete, as determined on the day after making, to be related to the compressive strength that would be reached by 28 days of normal curing. For the particular materials and variables tested, the relationship of accelerated to 28-day compressive strengths has proved to be:

$$R_{28} = \frac{26160\ R_a}{R_a + 11620}$$

where R_{28} is the compressive strength at 28 days of standard moist-cured cylinders and R_a is the corresponding accelerated strength.

2. Since the procedure takes into account the setting time of the concrete, variations due to different cements and admixtures are allowed for. Other variations in mix proportion, air contents etc. appear to have no effect. The effects of different aggregates have not yet been fully investigated. Any danger of low-strength aggregates can be determined at the mix design stage for particular materials.

3. A practical use of accelerated strength testing would be to provide the data needed for concrete quality control 27 days sooner than with the presently accepted procedure.

4. A testing program to establish fully the significance and reproducibility of accelerated strength testing in relation to a wide range of concretes should be undertaken. Further investigation of acceleration by hot or boiling water should include the effects of capping, temperature rise, aggregates, and other factors.

5. Acquaintance with accelerated strength testing suggests the need for a standard test procedure and raises the possibilities that such a procedure may in its own right satisfactorily measure concrete strength and quality so that reference to the conventional conception of 28-day compressive strengths may no longer be necessary. On this point it is hoped that concrete savants will feel challenged by the question "Why 28 days?"

Acknowledgments:

The work described in this paper was undertaken by the Materials and Research Division, Ontario Department of Highways. The authors' thanks are due to the staff who took part in the project.

The paper is presented by permission of H. W. Adcock, Assistant Deputy Minister (Engineering), and A. Rutka, Materials and Research Engineer.

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DISCUSSION

MR. BRYANT MATHER.[1]—The authors' discussion of alternatives to avoid overtime work left the impression that this problem remained unsolved. It is suggested that, when the volume of testing to be done is such as to cause a considerable amount of such overtime work on a particular concrete, the solution might be to place the cylinders in the tank during normal working hours and set a time clock to open a valve and admit boiling water at the appropriate time.

MR. V. M. MALHOTRA.[2]—The paper on the accelerated strength testing of concrete is another valuable contribution to the literature on this subject. The writer fully agrees with the authors regarding the desirability of an accelerated strength test and supports the idea that a coordinated action is needed.

In their introductory remarks the authors refer to the work of Patch, stating, ". . . full advantage does not appear to have been taken of his idea." The authors, however, ignore the fact that the U. S. Bureau of Reclamation discarded the test as proposed by Patch after several years of trial because of its unreliability. Since then various other organizations throughout the world have continued experimenting with one of the forms of the accelerated test but so far have failed to come up with any particular test which would be universally applicable. It should be of interest to mention that some of these organizations have test data which run into thousands of test results. The organizations that have experimented with the accelerated testing of concrete and of which the author has personal knowledge are: (1) Snowy Mountains Hydro-Electric Authority, Australia; (2) Sydney Water Board, Australia; (3) Irrigation Research Institute, Roorkie, India; and (4) Burrinjuck Dam Authorities, N.S.W. Australia.

In addition, several hydroelectric authorities in Italy, France, and Switzerland have been active in this field.

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[1] Engineer, Concrete Division, U. S. Army Engineer Waterways Experiment Station, Jackson, Miss.

[2] Scientific Officer, Construction Materials Section, Department of Mines and Technical Surveys, Ottawa, Ont., Canada.

Furthermore, The International Union of Testing and Research Laboratories for Materials and Structures (RILEM) is currently conducting a correspondence symposium, "Accelerated Hardening of Concrete With a View to Rapid Control Tests," in which the Mines Branch is one of the participants.

The writer has been engaged on accelerated strength testing of concrete off and on for the last six years, and had investigated the effect of time delay between mixing and curing on one form of accelerated strength test. The curing method used was as proposed by Akroyd and Smith-Gander, with certain modifications. The delay time was varied from ½ to 3 hr. The curing cycle consisted of placing the test cylinders 30 min after mixing in a water tank at 150 ± 5 F. The water was brought to boiling point within 1 hr and maintained at that temperature for the remaining curing period of 6 hr. The test cylinders were tested 8 hr after removal from the water tank. A delay period of 8 hr was selected in order to fit in with the shift work. The results of three concrete test series, with water-cement ratios varying from 0.45 to 0.65, involving 15 test mixes, are summarized in the accompanying Table 3. These results were then subjected to linear regression analysis, a summary of which is given below:

TABLE 3.—EFFECT OF DELAY BETWEEN MIXING AND CURING.

Water:Cement Ratio (by Weight)	Delay Between Mixing and Curing, hr	Compressive Strength,[a] psi	
		Accelerated Cured	28-Day Standard Cured
0.45	½	2140	5480
	1	1750	5110
	1½	1690	5070
	2	1470	4060
	3	1310	4630
0.55	½	1260	3650
	1	1520	3930
	1½	1540	4130
	2	1160	3490
	3	1230	3450
0.65	½	790	1980
	1	870	2540
	1½	970	2410
	2	790	2780
	3	740	2660

[a] Mean of three 6-by 12-in. cylinders.

Calculated correlation coefficient, r... = 0.641
Required value of r for correlation to be highly significant (from statistical tables).................... >0.641
Regression equation $Y = 39.4 - 29x$, where
x = time delay between mixing and curing, and
Y = accelerated cured strength as percentage of 28-day compressive strength.

Statistical studies indicated that the correlation between the time delay and the ratio of accelerated strength to 28-day compressive strength was "highly" significant and that strength decreased with increasing time delay. Unfortunately the accelerated method used by the writer and the method investigated by the authors are not similar and therefore the results cannot be compared directly. But this phenomenon of decrease in strength with increased time delay between mixing and curing is contrary to the authors' findings and should be further investigated. It should be pointed out that Fig. 2 in the paper under discussion is based upon the results of only one test mix involving one water-cement ratio and 15 test cylinders. It is doubtful whether satisfactory conclusions regarding relationship between accelerated strength and delayed curing time can be drawn from such limited test data.

The writer had further investigated the effect of delay between curing and testing time for the above accelerated strength test to see whether the testing time could be fitted in with shift change without involving overtime work. The results of three concrete test series, with water-cement ratios varying from 0.45 to 0.65, involving 21 test mixes, are summarized in Table 4.

Statistical studies showed that the correlation between the time delay between curing and testing and ratio of accelerated strength and 28-day strength was "highly" significant, and there is evidence that accelerated strength increases with increasing time delay.

TABLE 4.—EFFECT OF DELAY BETWEEN CURING AND TESTING.

Water: Cement Ratio (by Weight)	Delay Between Curing and Testing, hr	Compressive Strength,[a] psi	
		Accelerated Cured	28-Day Standard Cured
0.45	2	1980	5330
	4	2070	5250
	6	1870	4730
	7	1880	4950
	8	2140	5480
	9	2060	4880
	10	1970	4730
0.55	2	1370	3990
	4	1340	3510
	6	1500	3810
	7	1540	4030
	8	1260	3650
	9	1470	3450
	10	1460	3760
0.65	2	640	2140
	4	780	2200
	6	850	2530
	7	840	2500
	8	790	1980
	9	930	2380
	10	900	2510

[a] Mean of three 6 by 12-in. cylinders.

The authors report greater scatter of test results above an accelerated strength of 3000 psi than below. Akroyd had reported similar scatter, though he had used cube specimens. The writer in his investigations has also found a similar trend when using cylinder specimens and the accelerated curing procedure mentioned earlier. It therefore appears that a higher scatter of results beyond about 3000 psi is associated with accelerated strength testing irrespective of the method of curing and type of specimens used. If this is so, then this may prove to be the most undesirable limit of the accelerated curing methods.

The writer hopes that in the near future some cooperative effort will be undertaken by ASTM to standardize the accelerated curing method. The writer suggests that if such a program does materialize the following variables, in addition to those suggested by authors, should be investigated: (1) effect of delay between curing and testing, (2) effect of specimen size and type of cements, and (3) confidence in degree of prediction of 28-day strength based upon accelerated cured strength of 1, 2, or 3 test cylinders.

MESSRS. P. SMITH AND B. CHOJNACKI (*authors' closure*).—Mr. Mather, by one of his eminently sensible, practical suggestions, overcomes one of the major problems to the widespread acceptance of the accelerated strength testing procedure developed by the authors. They thank him for it and only labored the point of overtime work in the paper in order to present fairly the adverse sides of the procedure and to bring out the point that the solutions to this problem already advocated by others, such as prolonged delay in starting the accelerated curing, were not satisfactory.

Mr. Malhotra, by drawing attention to the forthcoming RILEM Symposium and to the interest being shown in accelerated strength testing in many faraway countries has usefully demonstrated that a real and universal desire for such a test probably exists. The authors felt that they had amply illustrated the different approaches already made to this same end by referring to those reports of previous work in England and North America which have been published.

That Patch's test method, and incidentally an even earlier one that has just come to the authors' notice,[3] did not meet with reliable acceptance in North America may now be explainable by the fact that such factors as the effect of delay time and hot-water curing time were not fully appreciated. In this respect, too, Mr. Malhotra may be missing the point in that the limited tests shown in Fig. 2 were only exploratory, and throughout the rest of the paper the effect of delay time and hot-water curing time are more fully examined as they relate to the kinds of concrete generally used in construction today. With such concretes often containing water-reducing admixtures, delay times of the order investigated by Mr. Malhotra are too short to avoid disruption of the concrete. A very low initial water bath temperature would be needed in conjunction with such delay times, and the accelerated strength then obtained would be of a much lower order.

The authors have not investigated the effect of time lapse between ending accelerated curing and testing the cylinders in compression. It is to be expected that residual thermal stresses on cooling and the moisture condition of the concrete would affect the results. Provided the time and cooling conditions are standardized, as in the tests reported in the paper, it is doubtful if those factors affect the validity of the over-all concept. Certainly along with the other unexplored factors listed by the authors and by Mr. Malhotra this is worthy of investigation if a method is to result from this or some other procedure in which such confidence can be placed that it can become a standard method of test.

The authors feel that the interest generated by this paper at the meeting and in written discussion has made the work that went into the project worthwhile, and they look forward to the concluding question "Why 28 days?", which they tossed into the stream of concrete thought, having ever widening ripples.

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[3] Gerend, "Steamed Cured Cylinders Give 28-day Concrete Strength in 48 hr.," *Engineering News-Record,* Vol. 98 (1927).

THE DEVELOPMENT OF PRECISION STATEMENTS FOR SEVERAL ASTM TEST METHODS*

BY S. J. HANNA[1] AND J. F. MCLAUGHLIN[1]

At present, relatively few standard methods of test contain information giving a quantitative indication of the agreement to be expected between "duplicate" runs, between operators, or between laboratories. A statement giving this type of information is commonly called a precision statement. The necessary components of a complete precision statement are:

1. The plus and minus limits for the variable, or a reasonable range for the data to lie within.
2. The confidence level at which these limits apply. Confidence level has been defined as "the long run probability of being correct when a certain type of assertation is repeatedly made" (**18**).[2]
3. The degrees of freedom associated with the determination of the plus and minus limits. Degrees of freedom are defined as "the number of observations (or classes, *sic*) which may vary independently of each other without necessarily altering the values of the constants estimated from the data" (**18**).

The primary purpose of this investigation was to illustrate an experimental design with appropriate analysis by which data could be collected to formulate precision statements for certain classes of ASTM test methods. Detailed procedures are given in the paper illustrating the general approach.

The approach used is a fundamental one. It begins with designing an experiment for the orderly collection of the important data needed to answer the pertinent questions and to provide the necessary information for the solution to a problem. An experimental design is a blueprint for the efficient collection of data for the purpose of answering specific questions or reaching specific objectives.

The analysis of the data is, in part, a consequence of the design. One asks the pertinent questions beforehand so the experimental model can be set up. Then the information or data necessary to the analysis can be obtained, and the investigator will be less likely to find himself with data that are useless for testing his hypothesis or, perhaps worse, insufficient data.

Three ASTM test methods were selected for study:

1. C 117, Method of Test for Materials Finer Than No. 200 Sieve in Mineral Aggregates by Washing,[3]
2. C 127, Method of Test for Specific Gravity and Absorption of Coarse Aggregate,[4] and
3. C 128, Method of Test for Specific Gravity and Absorption of Fine Aggregate.[3]

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* Presented at the Sixty-sixth Annual Meeting of the Society.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

[3] 1958 Book of ASTM Standards, Part 4.

[4] 1961 Book of ASTM Standards, Part 4.

Before any testing was done, the variables were determined and the experimental design model was established for each test method. Two variables present in each method were operators and materials. Sampling was not included as a variable in any of this work. The repeated tests were performed on the same sample of material or on prepared samples wherein the property being measured was controlled.

Statistical analyses of the data collected were made and from these, confidence limits and control limits were determined at the 95 and 99 per cent confidence levels. This investigation was limited to an illustration of the determination of the within-laboratory precision of the test methods for different operators following *exactly* the procedures for the methods as given in the ASTM standards. The general procedures illustrated in the text can be extended, however, to include additional levels of the variables under consideration or additional variables such as equipment laboratories.

STATISTICAL PROCEDURES

To accomplish the stated objective there are, generally speaking, many experimental designs that could be used. The choice of the model must be made by the experimenter, but the choice will necessarily be influenced by the information being sought, the flexibility, and the economy of the model. Careful study of the problem before testing has begun will eliminate the gathering of data that will be useless for the testing of the experimenter's hypothesis.

In keeping with this line of thought, each test method was examined to determine variables present, and a model for each method was selected so as to give the desired information. For the three methods investigated, the use of factorial models seemed to be advantageous. Ostle (12) in his discussion of factorial models states the following advantages and disadvantages:

Advantages:

1. Greater efficiency in the use of available experimental resources is achieved.
2. Information is obtained about the various interactions.
3. The experimental results are applicable over a wider range of conditions; that is, because the various factors are combined in one experiment the results are of a more comprehensive nature.
4. There is a gain due to the hidden replication arising from the factorial arrangement.

Disadvantages:

1. The experimental setup and the resulting statistical analysis are more complex.
2. With a large number of treatment combinations the selection of homogeneous experimental units becomes more difficult.
3. Certain of the treatment combinations may be of little or no interest; consequently, some of the experimental resources may be wasted.

It appeared that the advantages far outweigh the disadvantages.

In this investigation sampling variation was eliminated by performing the test a number of times on the same sample. Since different operators would be using the test method, operators were included as a random variable. Type of material was also included as a random variable.

For Method C 117, in addition to operators and types of materials, the amount of −No. 200 material was also included as a variable. The range from 1 per cent to 10 per cent of −No. 200 material was chosen as being of primary importance, and four levels were selected within this range. A description of the method of preparing these samples is included in the section on procedures.

Amount of −No. 200 material is considered to be a fixed variable since the extremes were represented and two other levels covered the range sufficiently.

The following is the mathematical model for Method C 117 – 49.[3] The mathematical models for Methods C 127 – 59[4] and C 128 – 57[3] are similar to this model except that there are two main effects rather than three. This naturally eliminates all terms containing the main effect symbol. The experimental error is then a function of three terms (replicates, main effect 1, and main effect 2).

Mathematical Model for Method C 117:

$$Y_{ijkl} = \mu + \rho_i + \alpha_j + \beta_k + \gamma_l + (\alpha\beta)_{jk} + (\alpha\gamma)_{jl} + (\beta\gamma)_{kl} + (\alpha\beta\gamma)_{jkl} + \epsilon_{ijkl}$$

where:

Y_{ijkl} = an individual observation in the ith replicate, in the jth level of factor a, in the kth level of factor b, and in the lth level of factor c,

μ = the true mean value,

ρ_i = effect of the ith replicate,

α_j = effect of the jth level of factor a (material),

β_k = effect of the kth level of factor b (amount),

γ_l = effect of the lth level of factor c (operators),

$(\alpha\gamma)_{jl}$ = effect of the interaction of the jth level of factor a with the lth level of factor c,

$(\alpha\beta)_{jk}$ = effect of the interaction of the jth level of factor a with the kth level of factor b,

$(\beta\gamma)_{kl}$ = effect of the interaction of the kth level of factor b with the lth level of factor c,

$(\alpha\beta\gamma)_{jkl}$ = effect of interaction of the three levels of factors,

ϵ_{ijkl} = effect of the experimental unit in the ith replicate to which the (jkl)th treatment combination has been randomly assigned,

the terms α_j, γ_l, $(\alpha\gamma)_{jl}$, adn ϵ_{ijkl} are assumed to be independently normally distributed with expected values of 0 and variances σ_α^2, σ_γ^2, $\sigma_{\alpha\gamma}^2$, and σ_ϵ^2 respectively, and

$$\sum_{i=1}^{r} \rho_i = \sum_{k=1}^{4} \beta_k = \sum_{k=1}^{4} (\alpha\beta)_{jk} = \sum_{k=1}^{4} (\beta\gamma)_{kl} = \sum_{k=1}^{4} (\alpha\beta\gamma)_{jkl} = 0$$

Prior to testing, an Analysis of Variance Table (ANOV) was formulated for each type method and the expected mean squares (EMS) were determined.

A Type I error, usually denoted by α, is committed if the hypothesis is rejected when it is actually true. Obviously one wishes to choose a test in which the probability of an error of this kind is small. It has been found generally acceptable in practice to use an α-level of 0.05, although this depends on the type of problem under consideration. For this study an α-level of 0.05 was used in performing F-tests.

The following equation was used in calculating the variance used in computing the control limits:

$$\text{Variance } (\bar{Y}_{jkl}) = \frac{\sigma_\alpha^2}{n_\alpha} + \frac{\sigma_\beta^2}{n_\beta} + \frac{\sigma_{\alpha\beta}^2}{n_{\alpha\beta}} + \frac{\sigma_\rho^2}{n_\rho} + \frac{\sigma_\epsilon^2}{n_\epsilon}$$

where:

σ_α^2 = variance component due to α,

σ_β^2 = variance component due to β,

σ_ρ^2 = variance component due to ρ,

$\sigma_{\alpha\beta}^2$ = variance component due to the interaction of α and β,

σ_ϵ^2 = variance component due to experimental error,

n_α = number of a's = a,

n_β = number of b's = b,

$n_{\alpha\beta}$ = a times b,

n_ρ = number of replicates = r, and

n_ϵ = total number of observations used to estimate variance, $\bar{Y}_{jkl}$, in this case abr.

The variance $\bar{Y}_{jkl}$ is used in computing control limits which are placed on the "grand" mean. If control limits are placed on means which are averaged over only one of the variables, then the appropriate variance, $\bar{Y}_x$, must be used. For example, if control limits are placed on means averaged over replicates only, the variance would be:

Variance $\bar{Y}_p = \sigma_p^2/n_p + \sigma_\epsilon^2/n_\epsilon$, where n is equal to the number of observations used to estimate the variance, in this case $n_\epsilon = n_p = r$.

PROCEDURES

The procedures detailed in the ASTM Standards were followed exactly by the operators when performing the tests. Two operators, acting independently, performed ten replicates for each test method investigated. The operators performed several practice tests to familiarize themselves with the method and test procedures before actual testing began.

From the data collected by the operators, sample means and sample variances were calculated. A statistical analysis was made on the data for each test method. Confidence limits were determined for 95 and 99 per cent confidence levels. From the statistical analysis of the data, precision statements of various kinds could be formulated for each test method studied.

Test Method C 117 – 49:

The purpose of Method C 117 – 49 is to determine the amount of material finer than the No. 200 sieve size in aggregate. Briefly the test consists of washing a tared sample of aggregate over a No. 200 sieve and then drying it to a constant weight and determining, by weight, the percentage lost.

The material used in the performance of this test was local concrete sand of glacial origin. The sand was prepared by washing it over a No. 200 sieve a number of times to eliminate all material finer than that sieve size. The sand was dried to a constant weight and material finer than the No. 200 sieve was added to batches of it in amounts so as to produce 500.0-g samples containing 1, 4, 7, and 10 per cent material finer than the No. 200 sieve. All test samples were prepared by a single individual and the −No. 200 material was weighed to the nearest 0.01 g.

Two types of −No. 200 material were used in the investigation. In one series limestone dust, obtained from commercial limestone filler, was used. In the other series, a limestone-residual clay soil was the source of the fine material.[5] The limestone dust was chosen so as to represent a noncohesive fine material and the clay was chosen so as to represent a very cohesive fine material.

For each replicate, eight samples were prepared and tested in random order. Each replicate contained samples representing the two material types and the four levels of each material. A table of random numbers was used to achieve the random order so as to eliminate any bias which might occur due to an ordered procedure of preparing and testing the samples. Replicates were made on different days to prevent any undue influence from the operator's mood on any one particular day from influencing the final results.

Test Method C 127 – 59:

The purpose of Method C 127 is to determine the 24-hr absorption and specific gravity of coarse aggregate. Briefly the test consists of immersing an oven-dried sample of coarse aggregate in water for 24 hr, then determining its weight in water and its weight (saturated surface dry) in air. The sample is then dried to a constant weight and weighed. The absorption and specific gravity are then calculated.

Two materials were used in this experiment. One was a crushed limestone from a quarry near Bloomington, Ind., the other was a local gravel. The coarse aggregates were graded so as to have equal amounts of material retained on the 1, $\frac{3}{4}$, $\frac{1}{2}$, $\frac{3}{8}$ in., and No. 4 sieves. They were sieved, washed, and dried to a constant weight, then the various fractions were combined to form a 5000.0-g sample.

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[5] Unified Soil Classification, CH.

For each replicate, the two samples were tested by each operator. Replicate determinations were made on different days to prevent bias. Upon completion of the testing, the two samples were again sieved and any change in weight of each fraction was noted.

Test Method C 128 – 57:

The purpose of Method C 128 is to determine the 24-hr absorption and specific gravity of fine aggregate. Briefly the method consists of soaking a tared sample for 24 hr and at the end of this period drying the sample to a saturated surface dry condition whereupon a 500.0-g sample is immediately placed in a 500-ml flask. Enough water is added to bring the volume to 500 ml and the weight of water added is determined. The specific gravities and absorptions are calculated.

The material used for this was local concrete sand. The sand was washed to remove material finer than the No. 200 sieve, dried to a constant weight, and separated into various fractions using a logarithmic series of sieves. Sand from these fractions was then recombined to form samples having two gradations, one with a fineness modulus of 2.40 and the other a fineness modulus of 3.00. Each sample weighed 1000.0 g.

Each of the two samples was then tested for absorption and specific gravity following the procedures outlined in Method C 128. One procedure not specifically noted in the ASTM standards was used, however. When the sample was removed from the 500-ml flask, the water was removed by decanting the sample. The sand plus the filter paper was then placed in the drying oven together and the dry weight obtained. The filter, paper was oven dried prior to testing to a constant weight and its dry weight obtained. By subtracting this weight from the combined dry weight the sample dry weight was obtained. This procedure was used to speed drying time; while the method does not explicitly state that the procedure should be followed, the wording does not preclude its use.

The portion of the sand not used in the flask was placed in a separate container and dried to a constant weight along with the sample from the flask. After the dry weights were determined the portions were recombined for the next replicate. As before, two operators acting independently performed ten replicates on each sample. One hundred per cent ethanol was used in minor amounts to reduce foaming in the flask.

Upon completion of testing, the two samples were sieved and the change in weight of each fraction noted. Loss in weight occurred during handling of the sample.

DISCUSSION OF RESULTS

The results of this study and the discussion of these results have been divided into four parts. The first part is concerned with the analysis of the data for Method C 117. The second part is concerned with the analysis of the data for Method C 127; the third part with the analysis of data for Method C 128; and the fourth part with the discussion of all test results and their application to the development of precision statements. The second and third parts have been further divided into two parts, one concerned with the analysis of absorption data, and the other concerned with the analysis of specific gravity data.

Results of Test Method C 117 – 49:

It should be recalled that a 3-factor factorial model was chosen for Method C 117. Operators and material were random variables and amounts of —No. 200 material was a fixed variable.

A Bartlett test for homogeneity of variance was performed on the data for operator A and resulted in accepting the hypothesis that the within-sample variances were equal. That is, the eight variances for two materials and four levels of each were accepted as being equal at an α-level of 0.05. The same conclusion was reached for the within-sample variances of operator B. However, a Bartlett test over the entire table of data (including both operators) resulted in the rejection of the hypothesis that all within-sample variances were equal at the 0.05 α-level.

With the problem of nonhomogeneous variance present, it was deemed advisable to analyze the results of operator A and operator B separately since one of the assumptions underlying the analysis of variance is that variances are homogeneous (**10**). Analyzing the data for operator A and operator B separately eliminated the operator variable and hence changed the statistical model from a 3-factor factorial to two 2-factor factorial models similar to the model for analysis of Method C 127.

The calculations for the sum of squares for the analysis of variance tables followed the procedure presented by Ostle (**12**, p. 351).

Table 1 is the Analysis of Variance Table for operator A and B, Method C 117. The estimate of experimental error, σ^2, for operator A is shown to be 0.0124. This mean square is used in testing hypotheses in regard to the effect of replicates, materials, and the operator-material interaction. The mean square for amounts was, as would be expected, very large, 296.1773, in comparison to

TABLE 1.—ANALYSIS OF VARIANCE TABLE FOR TEST METHOD C 117.

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	Expected Mean Square
		OPERATOR A		
Replicates...	$(r - 1) = 9$	$R_{yy} = 0.1819$	0.0202	$\sigma^2 + ab\,\sigma_\rho^2$
α........	$(a - 1) = 1$	$R_{yy} = 0.0594$	0.0594	$\sigma^2 + rb\sigma_\alpha^2$
β........	$(b - 1) = 3$	$B_{yy} = 888.5318$	296.1773	$\sigma^2 + r\sigma_{\alpha\beta}^2 + ra \sum_{K=1}^{K=4} \frac{\sigma_k^2}{(b - 1)}$
$\alpha\beta$........	$(a - 1)(b - 1) = 3$	$(AB)_{yy} = 0.0609$	0.0203	$\sigma^2 + r\sigma_{\alpha\beta}^2$
Experimental error.....	$(r - 1)(ab - 1) = 63$	$E_{yy} = 0.7851$	0.0124	σ^2
Total...	$(rab - 1) = 79$			
		OPERATOR B		
Replicates...	$(r - 1) = 9$	$R_{yy} = 0.0490$	0.0054	$\sigma^2 + ab\,\sigma_\rho^2$
α........	$(a - 1) = 1$	$A_{yy} = 0.0003$	0.0003	$\sigma^2 + rb\,\sigma_\alpha^2$
β........	$(b - 1) = 3$	$B_{yy} = 896.4696$	298.8232	$\sigma^2 + r\sigma_{\alpha\beta}^2 + ra \sum_{i=1}^{i=10} \frac{\beta_k^2}{(b - 1)}$
$\alpha\beta$........	$(a - 1)(b - 1) = 3$	$(AB)_{yy} = 0.0075$	0.0025	$\sigma^2 + r\,\sigma_{\alpha\beta}^2$
Experimental error.....	$(r - 1)(ab - 1) = 63$	$E_{yy} = 0.1738$	0.0028	σ^2
Total...	$(rab - 1) = 79$			

the variance which was used to test the hypothesis that $\sigma_\beta^2 = 0$ (amount effect). This effect must be tested using the mean square for amount-material interaction since the expected mean square for amount includes σ^2 and $\sigma^2_{\alpha\beta}$.

F-tests performed on the data from the analysis of variance indicated that materials and amounts were significant. That is, one cannot at the 0.05 α-level accept the hypothesis that $\sigma_\alpha^2 = 0$ (material effect) and that $\sigma_\beta^2 = 0$ (amount effect). The results of the F-tests indicated further that one can accept the hypothesis that $\sigma_r^2 = 0$ (replicate effect) and $\sigma^2_{\alpha\beta} = 0$ (material-amount interaction)

The estimate of experimental error for operator B was 0.0028. Since the expected mean squares for this design model are the same as those for the previously discussed model the same procedures of hypothesis testing were followed. As before the mean square for amounts was very large, 298.8232, with respect to the mean square used in testing the hypothesis that $\sigma_\beta^2 = 0$, namely, $\sigma^2 + \sigma^2_{\alpha\beta}$.

F-tests performed on the data from the analysis of variance of the data for operator B indicated that amounts were significant, $\sigma_\beta^2 \neq 0$, at 0.05 α-level.

Amounts would be expected to be significant since their means were chosen to be different. The fact that the material effect was significant for operator A indicated that the type of −No. 200 material has some effect on the results obtained. An examination of the means for operator A indicates that generally a higher percentage of −No. 200 material was determined for the finely ground limestone than for −No. 200 clay material (Table 2).

TABLE 2.—MEANS DETERMINED FOR DATA OF OPERATOR A.

Material	Limestone				Clay			
Per cent added....................	1	4	7	10	1	4	7	10
Per cent determined by Method C 117...........	1.066	4.052	7.006	10.030	1.080	3.912	6.952	9.988

The fact that material was found not significant for the data of operator B, coupled with the fact that the variances of operator A and operator B were found to be different, indicates that the results obtained using this test method are highly sensitive to the skill of the operator performing the test. Another factor lends some support to this suggestion. Operator B was an experienced operator while operator A was not (even though he was given a reasonable learning period). Therefore the results obtained by operator B may be indicative of results obtained when the test method is performed by an experienced laboratory technician, while the results obtained by operator A may be indicative of results obtained when the test method is performed by a person not trained in laboratory techniques of this nature.

The problem of establishing an acceptable range within which results of duplicate determination should agree, or establishing an interval within which one expects, with some given probability, the population parameter being estimated to lie, was complicated by the nonhomogeneity of variance encountered.

Another factor that arises is selecting the *type* of limits to be set, that is, confidence limits or control limits. Confidence limits on a mean or observation provide a range that, with a given prob-

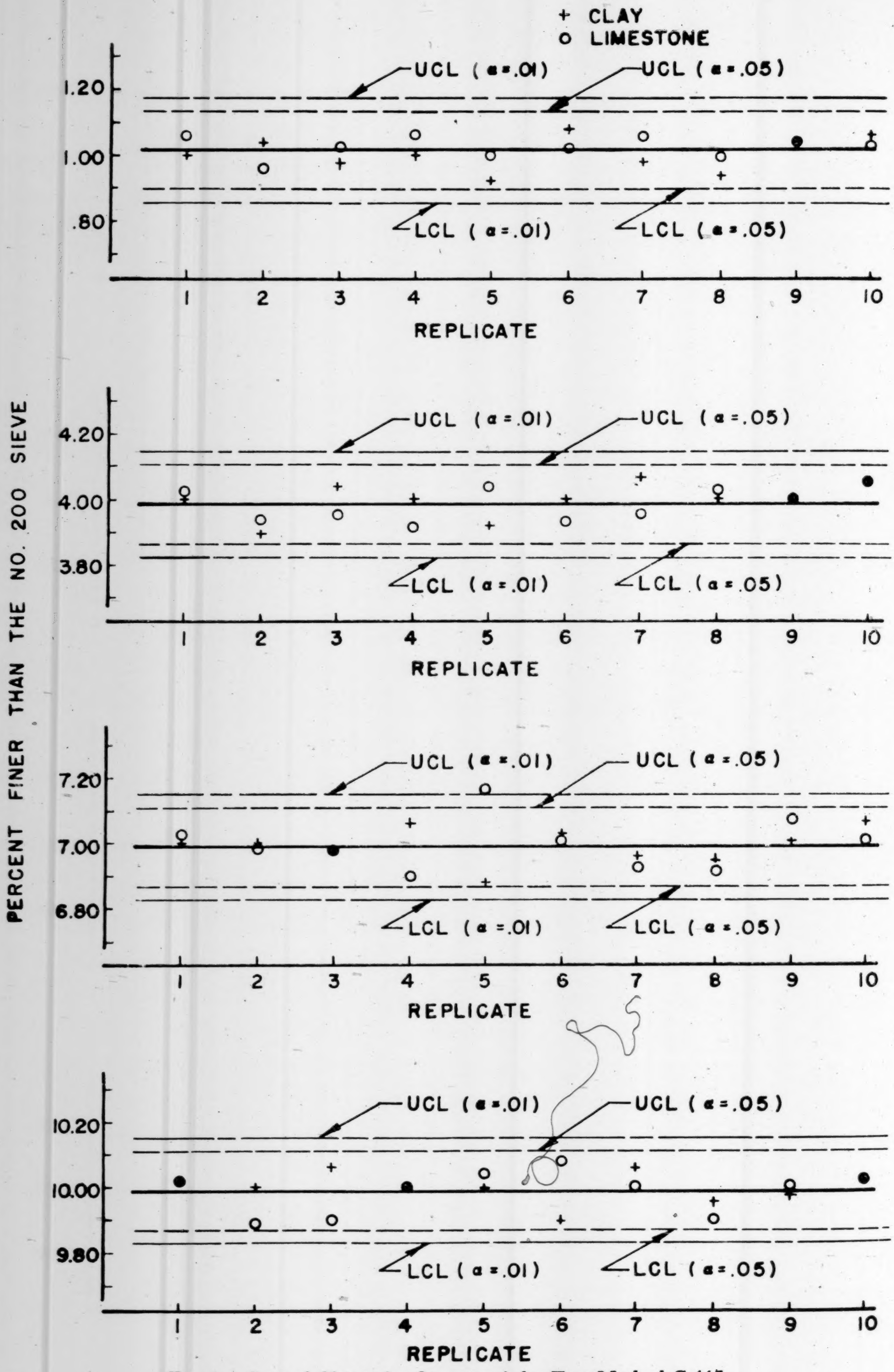

Fig. 1.—Control Charts for Operator A for Test Method C 117.

ability, include the true mean, μ. Control limits on the other hand provide limits in which a certain per cent of the sample statistics should fall in the long run. A set of data that has a number of observations outside the control limits (greater than that amount determined by choice of an α-level) is said to be out of control. Several factors may account for the data being out of control. One can be the fact that some assignable cause had led to discrepancies between observations, but another is that the precision of the test method or process is incapable of producing results of the quality required.

For the purpose of this study it seems appropriate to present both confidence limits and control limits. These limits are presented for the data of operator A and operator B separately.

Confidence Limits for Operator A.—From Table 1, $s^2 = 0.0124$; therefore $s = 0.111$.

Ninety-five per cent confidence limits on the mean of n observations made to determine the amount of material finer than No. 200 sieve are $\pm t_{0.05}\, s/\sqrt{n}$, where t is based on the chosen α-level (0.05 in this case) and the degrees of freedom associated with s. In this case the number of degrees of freedom is 63. For these conditions, the value of t is 1.998 and the limits are $\pm 0.222/\sqrt{n}$.

If, for example, three observations are made the confidence limits placed on the average of the three is $\bar{Y} \pm 0.222/\sqrt{3} = \bar{Y} \pm 0.128$. The interpretation that is placed on these limits is that one is 95 per cent confident that the true mean, μ, lies within the limits established by $\bar{Y} \pm 0.128$ where $\bar{Y}$ is the observed mean.

Control Limits for Operator A.—The establishment of control limits for a measurement process can give a basis for determining whether observations are made within the limits of variability allowable or inherent in the process. The control limits were placed on individual observations. Since both material and amount were determined significant, the limits were placed about the average of ten replicates. The limits, placed on individual observations are wider than limits placed on means. The limits are indicative of how well an operator may repeat his measurements.

The limits are as follows:

Estimate of variance

$$= \sigma^2{}_{Y_{jr}} = \frac{\sigma_\rho{}^2}{1} + \frac{\sigma^2}{1} = 0.0134$$

For 0.05 α-level based on n observations, where n equals ten replicates, $t_{0.05} = 2.262$. The control limits are then equal to: $\pm 2.262\ \sqrt{0.0134} = \pm 0.262$ per cent.

For 0.01 α-level, $t = 3.25$ and the control limits are: $\pm 3.25\ \sqrt{0.0134} = \pm 0.376$ per cent.

A plot of the observations in each replicate and the control limits as determined above are shown in Fig. 1 for the clay material. A similar chart can be constructed for the limestone material except that the mean, $\bar{X}$, would be shifted.

Confidence Limits for Operator B.—From Table 1, $s^2 = 0.0028$; therefore $s = 0.0548$.

Ninety-five per cent confidence limits equal $\pm 0.110/\sqrt{n}$ following the same procedures shown in the determination of confidence limits for operator A.

Control Limits for Operator B.—Control limits for data of operator B were placed on individual observations. The limits were plotted about means averaged over replicates and materials since amounts were significant. The procedures followed in determining these limits were the same as those used for determining control limits for the data of operator A except that obviously a different variance was used, that associated with

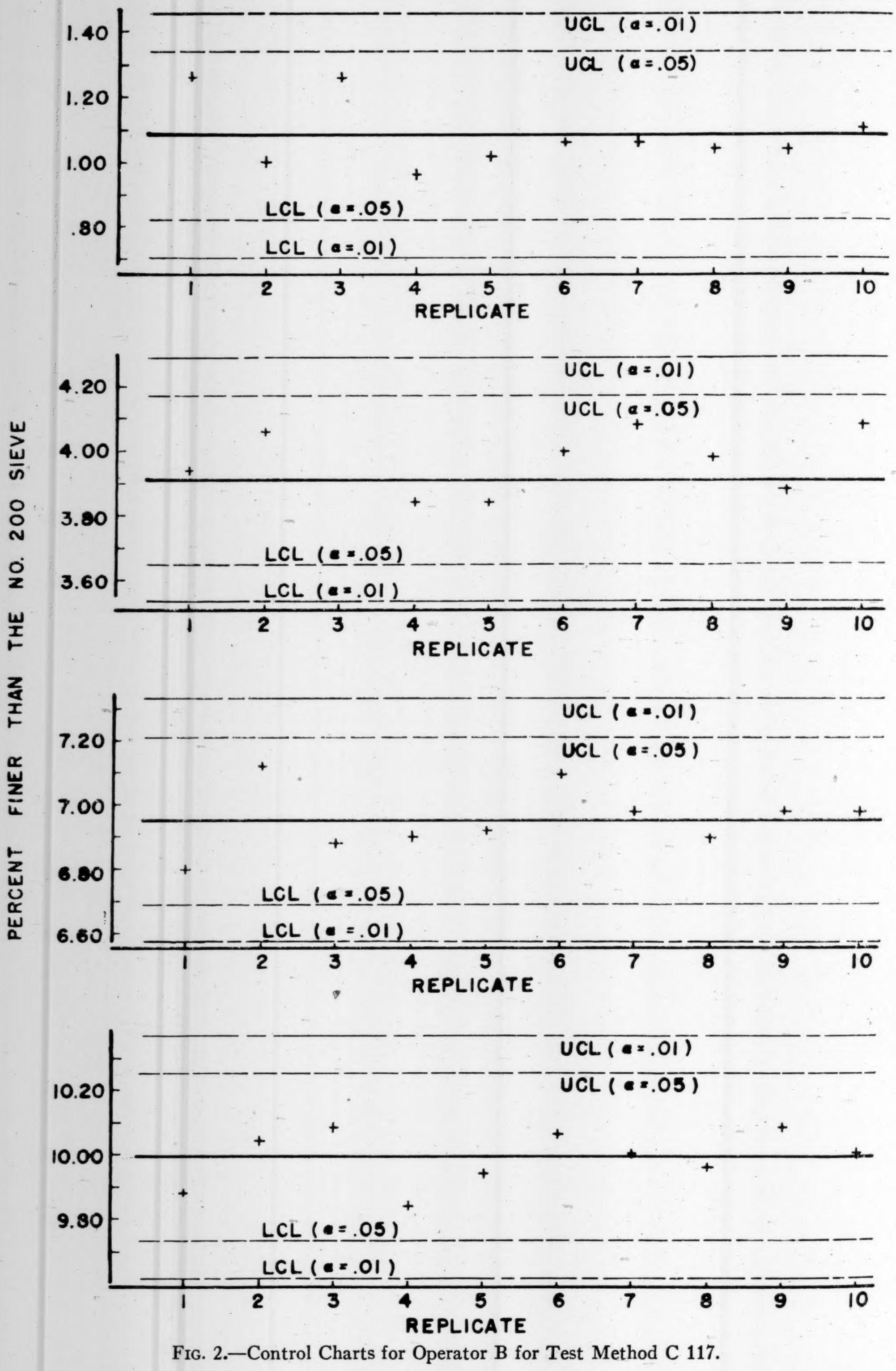

FIG. 2.—Control Charts for Operator B for Test Method C 117.

operator B. The limits are: ±0.116 per cent for an α-level of 0.05, and ±0.159 per cent for an α-level of 0.01.

Figure 2 shows the observations and the control limits as determined above.

General Observations.—Figures 1 and 2 give an indication of how wide the limits are and how some of the data were scattered. This scattering could be due to several factors (for example, a variable which was not controlled, such as laboratory humidity, may be significant). It is of interest to note that the ASTM committee responsible for this test method has revised it so that the weight determination and calculation of per cent finer than No. 200 sieve is made to the nearest 0.1 per cent instead of the nearest 0.02 per cent. The remainder of the revision appears to be in keeping with procedures followed during this study. If the per cent finer than No. 200 sieve was determined to the nearest 0.1 per cent, the variances are, for practical purposes, zero for the data of operator B. The situation is then similar to the situation encountered for the specific gravity data of Method C 127 – 59. Namely, the data are insensitive to statistical analyses and practical limits of ±0.1 per cent seem appropriate. Noting what happened in the case of operator A, the placement of these "practical" limits may not be justified.

TABLE 3.—ANALYSIS OF VARIANCE TABLE FOR ABSORPTION DATA FOR METHOD C 127.

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	Expected Mean Square
Replicates.....	$(r - 1) = 9$	$R_{yy} = 0.0128$	0.00142	$\sigma^2 + \sigma b \sigma_\rho{}^2$
α..........	$(a - 1) = 1$	$A_{yy} = 1.4745$	1.47450	$\sigma^2 + r\sigma^2_{\alpha\beta} + rb\sigma_\alpha{}^2$
β..........	$(b - 1) = 1$	$B_{yy} = 0.0084$	0.00840	$\sigma^2 + r\sigma^2_{\sigma\beta} + ra\sigma_\beta{}^2$
$\alpha\beta$..........	$(a - 1)(b - 1) = 1$	$(AB)_{yy} = 0.0130$	0.01300	$\sigma^2 + r\sigma^2_{\alpha\beta}$
Experimental error........	$(r - 1)(ab - 1) = 27$	$E_{yy} = 0.0394$	0.00146	σ^2
Total.....	$(rab - 1) = 39$			

Results of Test Method C 127 – 59:

Prior to testing, the variables present were determined and a 2-factor factorial model was chosen for the analysis of absorption and specific gravity data.

Absorption.—A Bartlett test for homogeneity of variance was performed and resulted in accepting the hypothesis that within-sample variances were equal at the 0.05 α-level. With these variances determined equal, it was then possible to proceed with the analysis of the data using the factorial model.

The sums of squares and the mean squares were calculated according to the procedures for 2-factor factorial models. These sums of squares and mean squares are shown in Table 3.

F-tests performed on the data from the analysis of variance indicated that there was a significant interaction between materials and operators, that is, $\sigma^2_{\alpha\beta} \neq 0$. Replicates and operators were not significant. The *F*-tests also indicated that materials were not significant, $\sigma_\alpha{}^2 = 0$. Materials were expected to be significant since aggregates having different absorptions were deliberately selected. The significant interaction can be logically explained. One operator may be more experienced, or more familiar, with one material than the other and this could conceivably influence his results.

With estimates of the variance components available, limits were determined. As before, both confidence limits and control limits were determined.

Control limits were placed on individual observations.

TABLE 4.—ANALYSIS OF VARIANCE TABLE FOR ABSORPTION DATA FOR TEST METHOD C 128.

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	Expected Mean Square
Replicates.....	$(r - 1) = 9$	$R_{yy} = 0.0896$	0.00995	$\sigma^2 + ab\sigma_\rho{}^2$
α..........	$(a - 1) = 1$	$A_{yy} = 0.2657$	0.2657	$\sigma^2 + r\sigma_{\alpha\beta}^2 + rb\sigma_\alpha{}^2$
β..........	$(b - 1) = 1$	$B_{yy} = 0.5108$	0.5108	$\sigma^2 + r\sigma_{\alpha\beta}^2 + ra\sigma_\beta{}^2$
$\alpha\beta$..........	$(a - 1)(b - 1) = 1$	$(AB)_{yy} = 0.0029$	0.0029	$\sigma^2 + r\sigma_{\alpha\beta}^2$
Experimental error	$(r - 1)(ab - 1) = 27$	$E_{yy} = 0.6096$	0.02258	σ^2
Total.....	$(rab - 1) = 39$			

The calculation of the components of variance results in $\sigma_\rho{}^2 = 0$. The sum of squares for replicates and experimental error were pooled to form a better estimate of the population variance. This variance was determined to be 0.00145 with 36 degrees of freedom. The control limits for $\alpha = 0.05$ and $\alpha = 0.01$ are indicated below.

For $\alpha = 0.05$.

$t_{0.05}$, 9 degrees of freedom $= 2.262$

control limits $= \pm 2.262\sqrt{0.00145/1}$

$= \pm 0.086$ per cent

For $\alpha = 0.01$.

$t_{0.01}$, 9 degrees of freedom $= 3.250$

control limits $= \pm 3.250\sqrt{0.00145/1}$

$= \pm 0.133$ per cent

Confidence limits were determined as follows: $\sigma^2 = 0.00145$ (pooled), therefore $\sigma = 0.038$. For this case, t equals 2.029 based on 0.05 α-level and 36 degrees of freedom. The confidence limits are therefore,

$$\pm 2.029\ (0.038/\sqrt{n}) = \pm 0.077/\sqrt{n}$$

Control limits were also determined for "between operators." These limits were determined as follows:

The estimate of variance, $\sigma_{\bar{Y}_j}{}^2$, equals 0.000073. The limits are $\pm t\sqrt{0.000073}$. For an α-level of 0.05 and 27 degrees of freedom, t equals 2.052 and the limits are ± 0.018 per cent. For an α-level of 0.01 and 27 degrees of freedom, t equals 2.771 and the limits are ± 0.024 per cent.

These limits are narrower than the control limits determined for a single operator and are applicable to situations where one is interested in making comparisons between *operator means*.

Specific Gravity.—A statistical analysis of the specific gravity data was not warranted since the range in values for each type of specific gravity never exceeded 0.02 and the variances were, for practical purposes, zero. The practical control limits for this case, therefore, are ± 0.01. These limits would also apply to between-operators since a check of the data indicated almost identical specific gravity determinations by each operator.

A precision statement as it might appear in the test method would be as follows: Duplicate determinations made by the same, or by different operators, should lie within the range determined by $\bar{Y} \pm 0.01$, where $\bar{Y}$ is the observed average of the specific gravity determinations.

One particular point was noted during the course of the study of Method C 127. This was that care should be taken, when submersing the sample into the water for the weight determination in water, to place the sample in the water in such a manner as to prevent as much as possible the entrapment of air. Possibly some reference in the test method is warranted.

Results of Test Method C 128 – 57:

Prior to testing, the variables present were determined and a 2-factor factorial model was chosen for the analysis of absorption and specific gravity data.

Absorption.—A Barlett test for homogeneity of variance was performed on the data. The test resulted in accepting the hypothesis that within-sample variances were equal at the 0.05 α-level. With these variances determined equal, it was then possible to proceed with the analysis of the data using the factorial model.

The sums of squares and the mean squares were calculated according to the procedures for 2-factor factorial models. These sums of squares and mean squares are shown in Table 4. The estimate of experimental error is 0.02258 as determined by the ANOV.

F-tests performed on the data from the analysis of variance indicated that operator effect was significant, $\sigma_\beta^2 \neq 0$, and also the material effect was significant, $\sigma_\alpha^2 \neq 0$. Replicate effect and the operator-material interaction were determined to be not significant.

Materials might be expected to be significant. Different fineness modulus (different gradations) were deliberately chosen. Since the material used was a natural sand of a heterogeneous nature, it is not unusual to find different size fractions having different absorptions.

It is interesting to note that operator effect was significant, indicating the sensitivity of the test method to this variable. The procedure involved requires judgment as to when a saturated surface dry condition has been obtained and it also involves some amount of skill on the part of the operator in placing 500.0 g of material into the flask quickly. It should be noted that only two operators were used in this study and therefore it is difficult to make generalities about the whole population of operators with confidence. This study does give an indication as to what might be expected.

With estimate of the variances components available, confidence and control limits were determined. To obtain an estimate of the precision of an operator the control limits were placed on the individual observations. The procedures followed in determining the control limits were the same as those used in determining the control limits for absorption data of Method C 127. For this case a pooled estimate of σ^2 was determined as 0.0190. Since $\sigma_\rho^2 = 0$ and $\sigma_{\alpha\beta}^2 = 0$, these were used with σ^2 to determine a pooled estimate of σ^2.

The pooled estimate of the variance σ^2 equals 0.0190 with 37 degrees of freedom. The control limits were determined as noted below.

For $\alpha = 0.05$

$$\text{control limits} = \pm 2.262\sqrt{0.0190/1} = \pm 0.312 \text{ per cent}$$

For $\alpha = 0.01$

$$\text{control limits} = \pm 3.25\sqrt{0.0190/1} = \pm 0.448 \text{ per cent}$$

Confidence limits were determined as follows. The pooled estimates of variance equal 0.0190, therefore σ equals 0.138. The 95 per cent confidence limits are, $\bar{Y} \pm 2.028\ (0.138/\sqrt{n}) = \bar{Y} \pm 0.280/\sqrt{n}$. The 99 per cent confidence limits are, $\bar{Y} \pm 2.716\ (0.138/\sqrt{n}) = \bar{Y} \pm 0.374/\sqrt{n}$.

Control limits were also determined for "between operators." These limits were determined calculating the variance and the degrees of freedom. The Satterwaithe equation was used to determine the degrees of freedom, which for this case was one. The control limits were determined as $\pm t\sqrt{\sigma_{\bar{Y}_j}{}^2}$. The $\sigma_{\bar{Y}_j}{}^2$ for this case was equal to 0.01325. The limits were then $\pm t\sqrt{0.01325}$ where t was based on the chosen α-level and the degrees of freedom determined, which was one. For an α-level of 0.05 the control limits are $\pm 12.706\sqrt{0.01325} = \pm 1.46$ per cent. These limits from a practical viewpoint are far too high. This points up the problems associated with trying to make generalities over a whole population with only one degree of freedom associated with the variance component due to operators. It should be further noted that the operator effect was determined significant, as previously mentioned, and would tend to indicate that the control limits would be wider for this case.

A comparison of the error variances of this test method with that determined for Method C 127 shows that this test method has a much higher component of variance due to experimental error than did Method C 127. These error variances were 0.0226 and 0.00146, respectively.

Specific Gravity.—Results of the analyses of variance performed on the data for the three types of specific gravity indicated that all have approximately the same magnitude of experimental error, namely, $\sigma^2 = 0.0001$. However, the estimate of this was difficult since the magnitude of computational figures are small and the factor of significant figures reduces the precision of the analysis. This estimate of σ^2 however, if used in setting control limits on means of duplicate determination (replicates) gives ±0.01. This is the same as the practical limits determined for the specific gravities of coarse aggregate determined by Method C 127. The specific gravities of fine aggregate as determined by Method C 128 are more variable than those of coarse aggregates as determined by Method C 127.

The variances associated with the specific gravity data were small, but some general observations may be made. Bulk specific gravity had more variance associated with it and is more sensitive to the operator variable and to the material variable than bulk specific gravity (SSD). Bulk gravity (SSD) in turn is more sensitive to the operator variable and the material variable than is apparent specific gravity. In fact, this study indicated no variance component due to the operator effect.

General Observations.—In the course of this study, it was noted that certain factors in Method C 128 could be improved or elaborated upon. The temperature bath specification contains no provision for any variance about 20 C. The time specified for the sample to remain in the bath also has no tolerance associated with it. The flask specified, 500 ml, is actually too small for the size of sample being tested.

The flask is not large enough to allow the sand to be mixed by the rolling action to remove entrapped air. It was noted that it was very difficult to remove the air entrapped in the sand at the bottom of the flask due to the fact that no mixture was achieved by the rolling action. A larger flask would provide sufficient room for the sample to be mixed with the water and the entrapped air released.

No provision is made for the counteraction of foaming that may occur during the removal of air by the procedure specified.

RESULTS APPLICABLE TO THE DEVELOPMENT OF PRECISION STATEMENTS

In the preceding sections, examples were shown of data collection and analysis and of the use of the results of these analyses for computing both confidence limits and control limits. The specific form of the precision statement must now be considered. In some cases, there may be no significant variability in the data whatsoever. In this case, a simple statement to that effect may be all that would be necessary. In other cases there may be only slight variability in the data and the results of the experiment would be insensitive to the statistical analysis (that is, the significant figures may be such that variances are much smaller than the number of significant figures in the data). In this second case, the use of the range would be sufficient to form the basis for a precision statement. The results of the specific gravity determinations fell into this category. Finally, there may be significant variation that does warrant the development of some type of plus and minus limit. Control limits appear to be the best way to express this information.

Control limits define a range of values which, in the long run, the sample of statistics will fall a certain percentage of the time. Further, these control limits indicate how well an operator is performing the test. If the operator's results are "out of control," some assignable cause may be the reason (for example, the operator may not be following exactly the test procedures specified).

For inclusion in a precision statement, control limits appear to be superior to confidence limits since they provide more information relative to the actual performance of the test method.

There are many forms in which precision information may be given. For example:

1. A simple statement of the repeatability and reproducibility determined for the test method.

   Example.—Duplicate determinations should check to the nearest 0.02 in the case of a single operator and to the nearest 0.05 for between-operators.

2. A statement giving the estimate of experimental error determined for the test method, the estimate of each variance component, and the degrees of freedom associated with each.

   Example.—The experimental error for the test method is 0.0015 with 27 degrees of freedom. The components of variance for replicates, operators, and materials are 0.0014 with 9 degrees of freedom, 0.008 with 1 degree of freedom, and 1.47 (highly significant) with 1 degree of freedom, respectively.

3. A statement giving the estimate of experimental error and the expression for computing control limits (for example, UCL = $\bar{Y} + t\sqrt{\sigma_{\bar{Y}}^2}$, $LCL = \bar{Y} - t\sqrt{\sigma_{\bar{Y}}^2}$ where $\bar{Y}$ = the sample mean and $\sigma_{\bar{Y}}^2$ = the variance about the mean). The degrees of freedom and the α-levels to be used with t must also be noted.

   Example.—The experimental error for the test method is 0.0015. Control limits are to be computed by $\bar{Y} \pm t\sqrt{\sigma_{\bar{Y}}^2}$, where $\sigma_{\bar{Y}}^2 = \sigma_{\epsilon}^2$ and t is based on an α-level of 0.05 and 9 degrees of freedom.

4. A statement giving the control limits for a single operator performing the test a number of times and the control limits for between-operators. The degrees of freedom and the α-level for determining t should also be given.

   Example.—Control limits for a single operator performing the test are $\bar{Y} \pm 0.03$, with 27 degrees of freedom associated with the estimate of experimental error and t based on 9 degrees of freedom and an α-level of 0.05. Control limits for between-operators are the same as for a single operator.

5. A statement giving the control limits only. The additional information on degrees of freedom and α-level could be footnoted to avoid confusion.

   Example.—Control limits for a single operator performing the test are $\bar{Y} \pm 0.03$.

Summary

The results of this study indicate that it is essential to determine the variables for a test method and to develop a statistical model before actual testing begins. The estimate of error variance and other variance components can be determined from an analysis of variance. With this information available, significance tests can then be made. Control limits can be placed on the data of a single operator and on data for between-operators.

In addition it was found that:

1. The experimental designs used produce information useful in determining control limits applicable to individual observations of the attribute being measured.
2. Control limits on individual observations form the basis for useful precision statements.
3. The data collected are also useful in the evaluation of the test method itself.

Precision Statements for Test Methods Studied:

A precision statement can be of great value to the user of the test. The limits provide a range of values in which the results of a duplicate test should lie with a certain probability. The operator is given a criterion for deciding if something has gone wrong. The variability of the test results, when compared with the expected variability, can give an indication of how well the test is being performed. That is, is the operator using the proper techniques and is he being accurate enough and careful enough in his measurements?

If the repeatability and reproducibility of the test method are known, it is then possible to compare results of different operators and laboratories on a rational basis. Errors that are not within the inherent error of the process or method can be discerned and the situation can be analyzed accordingly.

The following are examples of the *form* that precision statements might take using the results of this study. These statements are based on the previously stated assumptions and include components of variance due to replicates, operators, and material. This study specifically eliminated the component of variance due to sampling and other variables not mentioned. A statement to be published as a part of the test method should be more broadly based. The precision statements are for test data when the tests are performed by experienced personnel following exactly the procedures as outlined in the ASTM standards for the test method.

C 117, Method of Test for Materials Finer Than No. 200 Sieve in Mineral Aggregates by Washing:

Control limits for a single operator performing the test are $\bar{Y} \pm 0.12$ per cent, with 63 degrees of freedom associated with the estimate of experimental error and t based on 9 degrees of freedom and an α-level of 0.05.

C 127, Method of Test for Specific Gravity and Absorption of Coarse Aggregate:

Absorption.—Control limits for a single operator performing the test are $\bar{Y} \pm 0.09$ per cent, with 27 degrees of freedom associated with the estimate of experimental error and t based on 9 degrees of freedom and an α-level of 0.05. Control limits for between-operators are the same.

Specific Gravity.—Practical control limits for both a single operator and between-operators are $\bar{Y} \pm 0.01$, based on 40 observations.

C 128, Method of Test for Specific Gravity and Absorption of Fine Aggregate:

Absorption.—Control limits for a single operator performing the test are $\bar{Y} \pm 0.31$ per cent, with 27 degrees of freedom associated with the estimate of experimental error and t based on 9 degrees of freedom and an α-level of 0.05.

Specific Gravity.—Practical control limits for both a single operator and between-operators are $\bar{Y} \pm 0.01$, based on 40 observations.

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C-NOTCH IMPACT TEST FOR STEELS AT HIGH HARDNESSES*

By G. Steven[1] and J. R. Handyside[2]

Toughness has been defined as "the ability of a material to absorb energy without breaking." The need for toughness in tool steels is intimately connected with their end application. In forging-die tool steels, for instance, high stresses are present at elevated temperatures; in shear blades, the steel must be able to maintain a sharp edge without chipping; in lathe tools, particularly those used for interrupted cutting, a friction-heated cutting edge bears the brunt of repetitive impact; the toughness required of drills is related to torsion or to torsion combined with shock. Experience shows that a steel can be "tough" under one set of operating conditions yet be "brittle" when used in a different application, depending upon the applied stresses, the part geometry and size, and the rate of loading.

A test used to rate the toughness of steel must measure the product of load and deformation up to the fracture point. As shown in Fig. 1, the area under the load-deformation curve is divided into elastic work and plastic work. The elastic work is the recoverable distortion of the lattice after the load is removed; the plastic work is the integral of the applied load and the inelastic strain. Fracture occurs when the ductility of the material is exceeded.

Among the tests used to obtain meaningful comparisons of the toughness of tool steels are tension tests (**1**),[3] bend tests (**1–6**), torsion tests (**7–9**), torsion impact tests (**10–13**), unnotched impact tests (**14–16**), a notched-impact bend test (**17**), and a drop-weight impact test (**18**).[4] Many of these tests have been critically reviewed by Wilmes (**5**) and Clayton-Cave et al (**19**).

The tension test is useful only when fracture is preceded by an appreciable amount of plastic deformation, such as that which occurs in tool steels heat treated to hardnesses of about Rockwell C 42 to 54. At very high hardness, however, minor misalignments in the tension setup or slight surface imperfections can cause these steels to fail in a brittle manner at stresses well below their potential yield strength. Therefore, other tests had to be devised to measure the toughness of steels at very high hardness.

Rose (**20**) discusses the toughness concept in relation to the material to be tested and the test method used. Different test methods have characteristically different ratios of normal (cohesive) stress to shear stress. For the tension

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper.

[4] The drop-weight impact test consists of releasing a guided weight from a given height, transmitting the blow through a V-shaped anvil to either cylindrical or flat-bar specimens. If the specimen does not rupture, the weight is intercepted on the rebound and is then dropped from successively greater heights until fracture occurs. The drop-weight impact strength is expressed in inch-pounds absorbed energy at the failure point.

test, the principal stress (normal) is twice the maximum shear stress, but both stresses are equal in torsion and torsion impact. It is also known that the ratio of maximum fiber stress to shear stress is greater in specimens tested in unnotched impact than it is in specimens stressed in tension. When the impact specimen surface is notched, the normal stress is further increased in comparison with the shear stress. A steel heat treated to a given strength level will show brittle failure in testing if the cohesive strength is exceeded prior to the shear strength. Conversely, if the shear strength of the material is reached first, it will deform plastically and thus produce a tough fracture. Heat treating a steel to higher hardnesses creates greater resistance to indentation (plastic flow). Therefore, in high-hardness steels the shear strength is raised and a brittle cleavage-type failure is more likely to result during testing for material toughness. Thus, it is apparent that no one test can predict the life of tool steels under all service conditions.

DEVELOPMENT OF THE C-NOTCH IMPACT TEST

This investigation was limited to the following objectives:

1. To develop a test that would rate the over-all toughness of different tool steel classes when the individual steels are heat treated to high hardness (above Rockwell C 56). The test was to be restricted mainly to steel compositions and treatments that would produce only a small amount of plastic deformation at failure (microplastic strain). This limitation avoids toughness comparisons of materials with high yield strength and low ductility and those combining low strength and high ductility but having the same total impact energy absorption (curves 1 and 2 in Fig. 1).
2. To establish heat treatments of individual tool steels that yield optimum impact toughness at very high hardness.
3. To supplement alloy development and processing investigations aimed at improved tool steel properties.
4. To permit toughness comparisons to be made at elevated temperatures, particularly at the service temperatures of high-speed steels.
5. To establish a simple and inexpensive test procedure that would yield reasonably reproducible test results. The test should be adaptable to the present testing equipment and thereby be more readily accepted by other laboratories.

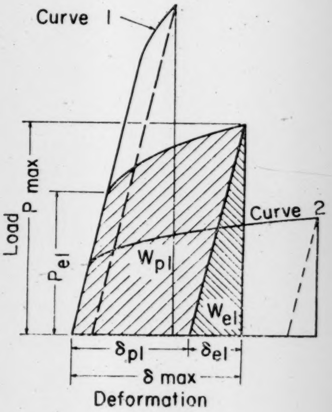


FIG. 1.—Schematic Load-Deformation Curves as a Measure of the Toughness of Steel.

P_{el} = Load at the elastic limit.
P_{max} = Fracture load.
δ_{el} = Elastic deformation.
δ_{pl} = Plastic deformation.
W_{el} = Elastic work.
W_{pl} = Plastic work.

After the reported test methods were reviewed, it was decided to develop a modified Charpy impact test. The test specimen was to contain a stress concentration factor, K_t, intermediate between that of the V-notch specimen (K_t = 3.9) and the unnotched specimen (K_t = 1). Modified Charpy impact specimens shown in Fig. 2 were machined with cylindrical notch radii of ⅛ in. (K_t = 1.55), ¼ in. (K_t = 1.31), ½ in. (K_t = 1.17), and 1 in. (K_t = 1.025).

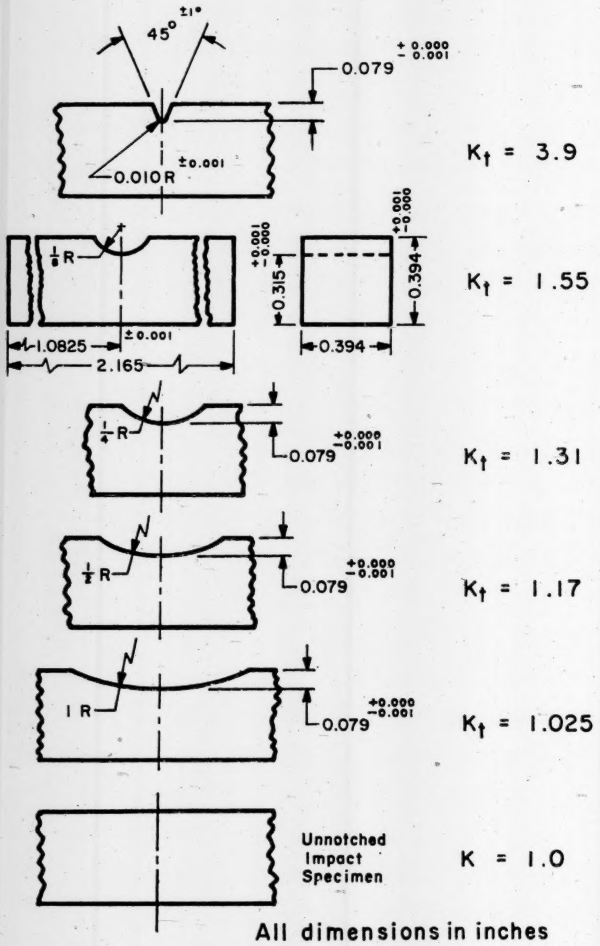

45°
±1°
0.079 +0.000 −0.001
0.010R
±0.001
Kt = 3.9
⅛R
+0.001 −0.000
0.315
+0.001 −0.000
0.394
1.0825
±0.001
2.165
0.394
Kt = 1.55
¼R
0.079 +0.000 −0.001
Kt = 1.31
½R
0.079 +0.000 −0.001
Kt = 1.17
1 R
0.079 +0.000 −0.001
Kt = 1.025
Unnotched Impact Specimen
K = 1.0
All dimensions in inches

FIG. 2.—Modified Charpy Impact Specimens with Notch Radius 0.010 (standard), ⅛, ¼, ½, 1 in, and Unnotched.

A preliminary evaluation was made on a conventional Charpy impact testing machine using four representative commercial tool steels: (1) an air-hardening steel, A-2; (2) a shock-resistant steel, S-5; (3) a high-speed steel, M-2; and (4) a hot-work steel, H-12, with a nominal composition of 0.35 per cent carbon, 0.35 per cent manganese, 1.05 per cent silicon, 5.00 per cent chromium, 0.35 per cent vanadium, 1.25 per cent tungsten, 1.35 per cent molybdenum. The latter two steels were tested at room temperature and at 1000 F. Figure 3 shows the Charpy impact resistance of these steels as a function of notch radii. The stress concentration factors of the notches are also plotted in this graph.

The data show that the impact strength for all steels increases with increasing notch radius. Furthermore, the sharpest rise in energy absorption occurs between a notch radius of about 0.010 (Charpy V-notch test) and 0.500 in. Because of the self-imposed limitations of minimum hardness, Rockwell C 56, and low plasticity for the test, the hot-work steels were excluded from all but one toughness comparison. In this instance, the low transverse impact properties of specimens machined from a large bar pointed toward the use of the C-notch specimen configuration. In general, however, hot-work steels with sufficient reduction from the ingot can be rated satisfactorily with the conventional V-notch impact test. Thus, the modified Charpy impact test should be considered as an extension of the V-notch impact test into a region of toughness where the latter is not sufficiently selective.

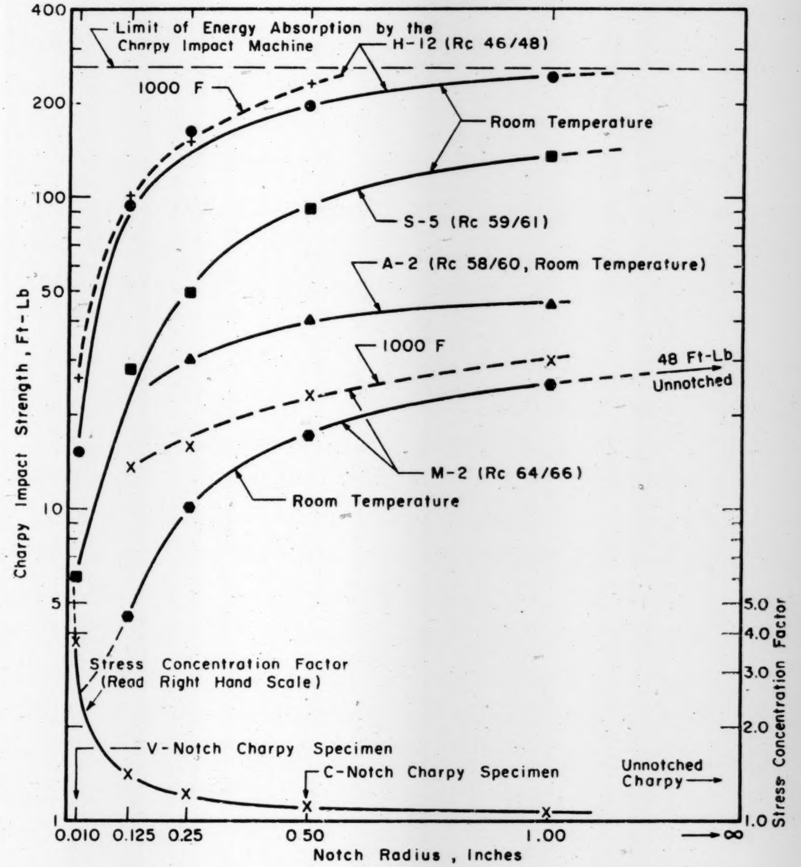


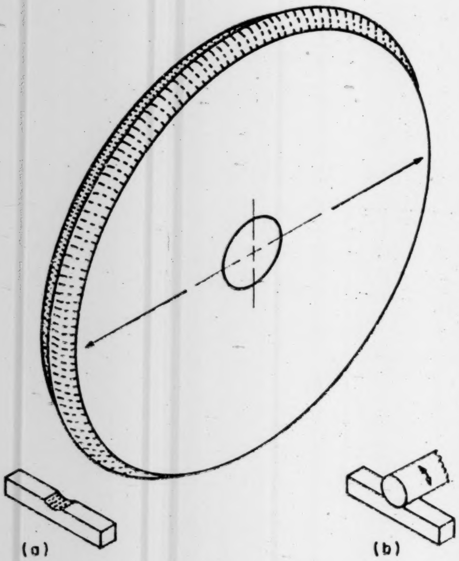
FIG. 3.—Effect of Notch Radius on the Charpy Impact Strength of Four Representative Tool Steels.

Stress concentration factor as a function of notch radius.

Based on the data in Fig. 3, a $\frac{1}{2}$-in. notch radius was selected to carry out a comprehensive toughness evaluation of tool steels. The term C-notch impact test henceforth refers to this standardized specimen geometry. Preliminary test results for different steels using the new specimens were reported in two prior publications. (21,22).[5]

A sequence of processing steps has been established for C-notch test specimens to ensure reproducibility of test results.

With the exception of the above noted tests on hot-work steels, all steels were forged to $\frac{5}{8}$- or $\frac{3}{4}$-in. square bars and rough machined into 0.430 by 0.430 by 2.2-in. blanks. Subsequent to heat treatment, the blanks were ground on all sides to the finished dimensions (Fig. 2).



(*a*) Transverse contour grinding the $\frac{1}{2}$-in. radius notch, with 8 in. by $\frac{3}{4}$-in. $1\frac{1}{4}$-in. abrasive wheel.

(*b*) Removing the transverse grinding marks by polishing the notch with a $\frac{3}{4}$-in. diameter medium India stone.

FIG. 4.—Method of Generating the Cylindrical Segment Notch in Modified Charpy Impact Specimens.

The notch radius was then ground into the hardened specimens with a dressed contour grinding wheel, and the transverse grinding marks were removed by honing circumferentially with a $\frac{3}{4}$-in. medium India stone (Fig. 4). The specimens were tested on a Riehle impact testing machine using the 240 ft-lb range (60-lb hammer, 4-ft drop height).

MATERIAL

Bar stock was selected from commercial and experimental heats of steel in each of the following tool steel classes: oil-hardening and air-hardening cold-work steels; shock-resisting steels; and high-speed steels. The chemical compositions of these steels are listed in Table I. The compositions of two cobalt-base cutting tool materials and two cobalt-bonded tungsten carbides are listed in Table VI.

EXPERIMENTAL RESULTS

Tables II and III list the C-notch Charpy impact data for cold-work steels tested in this program. Specimens from the different tool steel types were austenitized at their conventional austenitizing temperatures and then oil quenched or air cooled to room temperature and tempered to different hardness levels (Rockwell hardness, C 56 min). All steels were then tested at room temperature; selected grades were also tested at −100 and 300 F. At a given hardness level, there is a gradual increase in impact toughness with increasing testing temperature from subzero to 300 F. When the tool steels are tempered between 300 and 1050 F, the C-notch impact resistance rises at first with increasing tempering temperature (decreasing hardness). The impact toughness then passes through a minimum or at least remains constant before resuming the rise. This is illustrated in Fig. 5 with impact curves for A-2 (sulfurized), A-6

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[5] In 1946, Grossmann (**23**) used Charpy impact specimens with 0.079-in. and 0.250-in. radii to determine the effect of testing temperature on absorbed energy in breaking hardened SAE 4150 steel.

TABLE I.—CHEMICAL COMPOSITIONS.

Steel	AISI Type Designation	Element, per cent								
		Carbon	Manganese	Sulfur	Silicon	Chromium	Vanadium	Tungsten	Molybdenum	Cobalt
OIL-HARDENING COLD-WORK STEELS										
1	O-1	0.9	1.2	...	0.3	0.5	...	0.5	...	...
1A	O-1	0.86	1.3	...	0.3	0.5	...	0.4	...	...
2	O-6	1.50	0.8	...	1.0	...	...	...	0.3	...
3	L-6[a]	0.73	0.7	...	0.2	0.8	...	...	0.3	...
4	L-7	1.05	0.4	...	0.3	1.2	...	...	0.3	...
5	Experimental High Strength Steel	0.57	0.2	...	2.0	1.3	0.3	...	0.4	...
AIR-HARDENING COLD-WORK STEELS										
6	A-2	0.98	0.7	...	0.4	5.2	0.3	...	1.1	...
7	A-2 Sulfurized	0.94	0.6	0.15	0.3	5.3	0.3	...	1.2	...
7A		0.99	0.7	0.15	0.3	5.3	0.3	...	1.2	...
8	A-6 Sulfurized	0.70	2.0	0.08	0.3	1.1	...	...	1.4	...
9	D-2	1.47	0.3	...	0.4	12.0	0.9	...	0.8	...
10	D-2 Sulfurized	1.45	0.3	0.12	0.4	10.8	0.8	...	0.8	...
10A		1.52	0.3	0.12	0.5	11.6	1.0	...	0.8	...
10B		1.54	0.3	0.13	0.4	11.4	1.0	...	0.8	...
11	D-4	2.18	0.4	...	0.5	11.4	0.2	...	0.9	...
12	Experimental Steel	0.94	2.3	...	0.6	1.4	...	...	1.1	...
13	Experimental Steel Sulfurized	0.96	2.5	0.13	0.4	1.0	...	...	1.2	...
SHOCK-RESISTING STEELS										
14	S-5	0.54	0.8	...	1.9	0.2	...	...	0.3	...
15	0.45C-Mn-Si-Cr-V-Mo Steel	0.43	1.3	...	2.1	1.5	0.3	...	0.4	...
16	L-2	0.54	0.8	...	0.3	0.9	0.2	...	...	...
HIGH-SPEED STEELS (MOLYBDENUM-BASE)										
17	M-2	0.81	0.3	...	0.3	4.3	2.0	6.0	5.0	...
17A		0.85	0.3	...	0.3	4.0	1.9	6.4	5.0	...
17B		0.84	0.3	...	0.3	4.1	1.9	6.4	4.9	...
17C		0.84	0.3	0.017	0.3	4.1	1.9	6.3	4.9	...
17D		0.85	0.3	0.011	0.4	4.1	1.9	6.2	4.9	...
17E		0.84	0.3	0.008	0.3	4.1	1.9	6.2	5.0	...
18	M-2S	0.84	0.3	0.12	0.3	4.2	2.0	6.2	5.1	...
18A		0.84	0.3	0.09	0.3	4.2	2.1	6.4	4.9	...
19	1.0C M-2S	0.96	0.3	0.09	0.3	4.2	2.0	6.2	4.9	...
20	1.0C M-2	0.99	0.3	0.03	0.3	4.1	2.0	6.3	5.1	...
21	M3S-1	1.02	0.3	0.13	0.3	4.0	2.3	6.3	6.2	...
22	M3S-2	1.19	0.3	0.10	0.2	4.0	2.8	6.4	6.1	...
23	M-7	1.00	0.3	...	0.2	3.9	2.2	1.7	8.5	...
23A		1.01	0.3	...	0.2	3.9	2.2	1.6	8.7	...
24	High C Type M-35	1.10	0.4	...	0.3	4.2	1.9	6.9	3.7	5.1
24A		1.09	0.3	...	0.4	4.3	1.9	7.0	3.8	5.3
25	1.5–3.5-1-4	0.85	0.3	...	0.3	3.2	1.2	1.5	4.0	...

TABLE I.—*Concluded.*

Steel	AISI Type Designation	Element, per cent								
		Carbon	Manganese	Sulfur	Silicon	Chromium	Vanadium	Tungsten	Molybdenum	Cobalt
		HIGH-SPEED STEELS (TUNGSTEN-BASE)								
26	T-1	0.72	0.3	...	0.3	4.0	1.1	17.8	...	...
26A		0.78	0.3	...	0.4	3.9	1.0	18.1	...	...
27	T-4	0.70	0.3	...	0.4	3.8	1.0	18.2	0.7	4.8
28	T-5	0.75	0.3	...	0.4	3.8	1.9	17.0	0.5	8.0
29	T-6	0.75	0.3	...	0.4	3.9	1.9	18.8	0.7	12.0
30	T-8	0.79	0.3	...	0.4	3.9	1.9	13.8	0.9	5.2
31	T-9	1.18	0.3	...	0.4	3.8	3.7	18.0	0.6	...
		HOT-WORK STEELS								
32	0.5C-8Cr-W-Mo	0.47	0.4	...	1.2	8.4	0.1	1.2	1.5	...
33	0.5C-8Cr-V-Mo	0.49	0.4	...	1.2	7.7	1.4	0.1	1.7	...
34	0.5C-5Cr-V-Mo	0.49	0.4	...	1.3	4.9	1.1	0.05	1.6	...

[a] Contains 1.4 per cent nickel.

(sulfurized), L-6, and S-5. The toughness minimum or toughness dwell of the relatively low-alloy L-6 and S-5 occur between 450 and 600 F tempering temperature. The toughness minimum is displaced to appreciably higher temperatures in the more highly alloyed A-2 (sulfurized) and A-6 (sulfurized). These observations were confirmed by Bungardt et al (15). These investigators show a shift in the embrittlement valley of high-hardness steels depending on the total alloy content. Specifically, composition X165CrV12, a D-2 type steel, when air cooled from 1020 C (1865 F) and tempered at a series of temperatures shows declining unnotched Charpy impact values between 200 and 400 C (392 to 752 F).

A comparison of the cold-work steels, heat treated as shown in Tables II and III, to the commercially important hardness ranges, Rockwell 61 to 63 and Rockwell 58 to 60, gives the following ranking of C-notch impact strength:

	Hardness Ranges[6]	
	R_c 61-63	R_c 58-60
Lowest	O-6, D-4	O-6, D-4
Intermediate	L-7 D-2 (sulfurized)	O-1 L-7 D-2 (sulfurized)
Best	A-2 (sulfurized) A-6 (sulfurized) L-6, O-1	A-2 (sulfurized) A-6 (sulfurized) L-6

The shock-resistant steels in Table IV show several times greater resistance to impact than the cold-work steels at Rockwell C 60 or below. It will be noted that the impact toughness of the 0.45C-Mn-Si-Cr-V-Mo steel does not drop at subzero temperatures.

Tables V and VI list the C-notch impact strength of some commercial high-speed steels at both room tempera-

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[6] In this connection it should be emphasized that the ranking of toughness among these steels can be changed depending upon the austenitizing temperatures and the hardness levels of the comparison.

TABLE II.—COLD-WORK OIL-HARDENING STEELS.[a]

Steel	AISI Designation	Heat Treatment		Rockwell Hardness, C-Scale	C-Notch Charpy Impact Energy, ft-lb, at Indicated Test Temperature					
		Austenitized	Tempered		−100 F	Average	Room Temperature	Average	300 F	Average
1......	O-1	1475 F, 1 hr	300 F, 2 hr	64	...		10, 12, 9, 14, 16, 23,	14	...	...
1A.....	O-1	1475 F, 1 hr	350 F, 2 hr	62	19, 21, 17	19	28, 28	28	38, 39	38
1......	O-1	1475 F, 1 hr	400 F, 2 hr	61	14, 17, 19, 21,	19	32, 35, 36, 38, 28, 26,	30	34, 47	41
1A.....	O-1	1475 F, 1 hr	400 F, 2 hr	61	19, 21		33, 24, 27, 28			
1......	O-1	1475 F, 1 hr	500 F, 2 hr	58	24, 16, 10, 21,	19	27, 28, 27, 41, 26, 40,	30	46, 35	40
1A.....	O-1	1475 F, 1 hr	500 F, 2 hr	58	21, 18		25, 26, 34			
1......	O-1	1475 F, 1 hr	600 F, 2 hr	56	28, 18, 12, 40,	27	37, 25, 20, 52, 47, 23,	32	37, 46	41
1A.....	O-1	1475 F, 1 hr	600 F, 2 hr	56	34, 27		30, 30, 26			
1A.....	O-1	1475 F, 1 hr	Austempered	56	36, 27	31	64, 62	63	73, 64	68
			500 F, 3 hr	...	...	...	...	...	...	...
2......	O-6	1550 F, 1 hr	300 F, 2 hr	63	...	...	1, 1, 4, 5	3	...	...
			500 F, 2 hr	60	...	...	10, 10, 11, 12	11	...	...
			600 F, 2 hr	58	...	...	8, 11, 12, 13	11	...	...
3......	L-6	1550 F, 1 hr	300 F, 2 hr	63	10, 10, 9	10	14, 17, 12, 12, 19	15	...	...
			400 F, 2 hr	60	20, 21, 18	20	33, 41, 44, 46, 51	43	...	...
			500 F, 2 hr	58	18, 16, 18	17	43, 50, 32, 38, 24	37.5	...	...
			600 F, 2 hr	56	17, 24, 23	21	82, 53, 81, 42, 82	68	...	...
4......	L-7	1525 F, 1 hr	350 F, 2 hr	63	...	...	15, 15, 16	15	...	...
			400 F, 2 hr	60	...	...	18, 26, 19, 19, 17	19	...	...
			500 F, 2 hr	59	...	...	22, 17, 21	20	...	...
			600 F, 2 hr	57	...	...	18, 24, 20, 15, 29	21	...	...
			900 F, 2 hr	47	...	...	27, 48, 52, 35, 45	41	...	...
5......	Experimental Steel	1700 F, 1 hr	600 F, 2 hr	60	135, 142, 155	144	161, 171, 181	171	...	...

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[a] All steels were oil quenched from the austenitizing temperature.

TABLE III.—COLD-WORK AIR-HARDENING STEELS.[a]

Steel	AISI Designation	Heat Treatment		Rockwell Hardness, C-Scale	C-Notch Charpy Impact Value, ft-lb, at Indicated Test Temperature					
		Austenitized	Tempered		−100 F	Average	Room Temperature	Average	300 F	Average
6.....	A-2	1750 F, 1 hr	350 F, 3 + 3 hr	61	...	...	22, 36, 31,	30	...	...
			600 F, 3 + 3 hr	58	...	...	40 33,35	36	...	...
7.....	A2—Sulfurized	1775 F, 1 hr	350 F, 3 hr	63	...	...	19, 12, 14, 13, 18	15	...	...
			400 F, 2 hr	61	12, 11, 16	13	16 tests (19–44)	31.5	...	...
			500 F, 2 hr	60	24, 28, 31	27	43, 45, 34	41	56, 61, 61, 64	61
			600 F, 2 hr	59	22, 18, 29	23	30, 40, 40	37		
			700 F, 2 hr	58	22, 22, 16	20	32, 31, 35	33	25, 29, 30, 36	30
			900 F, 2 hr	58	17, 24, 21	21	23, 32, 32, 30, 27	29	40, 41, 41, 45, 44, 45, 46	43
7A....	A2—Sulfurized	1800 F, 1 hr	400 F, 2 + 2 hr	61	13, 19, 19, 16	17	19, 23, 29, 32	26	...	...
			400 F, 3 hr	61	17, 20, 22, 23	21	27, 28, 31, 41	32	...	...
			650 F, 3 hr	60	23, 25, 27, 29	26	39, 41, 41, 43	41	...	...
			700 F, 2 + 2 hr	58	...	...	24, 25, 25, 31	26	...	...
			900 F, 2 + 2 hr	59	...	...	12, 16, 16, 25	17	...	...
			1050 F, 3 hr	56	30. 36, 36, 41	36	39, 44, 44	42	...	...
8.....	A6—Sulfurized	1600 F, 1 hr	300 F, 2 hr	62	10, 12, 8	10	24, 15, 14, 13, 35	20	...	...
			400 F, 2 hr	59	24, 18, 12	18	16 tests (28–56)	42	...	...
			500 F, 2 hr	58	45, 38, 26	36	67, 70, 52, 63, 59	62	...	...
			600 F, 2 hr	56	45, 34, 43	41	66, 76, 60	67	...	...
			1000 F, 2 hr	50.5	...	...	60, 45, 45, 48, 53	50	...	...
			1050 F, 2 hr	48	...	...	75, 76, 83, 51, 83	74	...	...
9.....	D-2	1850 F, 1 hr	350 F, 3 + 3 hr	63	...	...	11, 13, 18	14	...	...
			600 F, 3 + 3 hr	59	...	...	23, 25, 22	23	...	...
10....	D-2—Sulfurized	1850 F, 1 hr	250 F, 3 + 3 hr	64	...	...	17 tests (11–18)	16.7	...	...
10A...			250 F, 2 + 2 hr	64	11, 11, 13	12	15, 18, 20	18	22, 17, 10	16
10B...			300 F, 2 hr	64	...	...	19, 18, 23, 15, 16, 15	17	...	...
10B...			500 F, 2 hr	60	...	...	15, 32, 20, 20, 19, 36	23	...	...
10A...			600 F, 2 hr	59	...	...	21, 21, 15, 23, 25, 22	21	...	...
10B...			700 F, 2 hr	59	...	...	26, 18, 19, 15, 20, 22	20	...	...
10B...			900 F, 2 hr	60	...	...	26, 24, 19, 28, 17, 14	21	...	...
10B...			1000 F, 2 hr	61	...	...	23, 27, 14, 14, 16, 19	19	...	...

11....	D-4	1825 F, 1 hr	350 F, 2 + 2 hr	63	...	...	11, 12, 12, 10	11	...	...
			400 F, 2 + 2 hr	61	...	...	12, 12, 12, 10, 10	11	...	...
			500 F, 2 + 2 hr	60	...	...	10, 9, 11, 8, 12	10	...	...
			700 F, 2 + 2 hr	59	...	...	10, 10, 10, 13, 11	11	...	...
			900 F, 2 + 2 hr	59	...	...	6, 7, 8, 7, 9	7	...	...
			1000 F, 2 + 2 hr	56	...	...	9, 10, 6, 8, 8	8	...	...
		1850 F, 1 hr	350 F, 2 + 2 hr	63	10, 10, 10	10	11, 12, 12, 10	11	13, 15, 11, 14	13
12....	Experimental Steel	1500 F, 1 hr	200 F, 1 + 1 hr	64	...	...	15, 16, 17, 16, 17	16	...	...
			400 F, 1 + 1 hr	60	...	...	17, 18, 19, 17, 18	18	...	...
			600 F, 1 + 1 hr	58	...	...	13, 23, 26, 16, 20	20	...	...
13....	Experimental Steel Sulfurized	1500 F, 1 hr	200 F, 1 + 1 hr	64	...	...	9, 9, 10, 10, 9	9	...	...
			400 F, 1 + 1 hr	60	...	...	14, 15, 16, 16, 17	16	...	...
			600 F, 1 + 1 hr	58	...	...	16, 17, 22, 16, 20	18	...	...

[a] All steels were air cooled from the austenitizing temperature.

ture and 900 F. As a group, the molybdenum-base high-speed steels have better impact toughness than the tungsten-base high-speed steels. The average C-notch impact resistance of conventionally heat-treated molybdenum-base high-speed steels used for general machining operations ranges between 9.5 and 20 ft-lb at room temperature and 14 to 35 ft-lb at 900 F testing temperature. The corresponding ranges of C-notch toughness for the tungsten-base steels is 3 to 12 ft-lb and 5 to 17 ft-lb, respectively, at the high temperature. There is a tendency for cobalt to lower the attainable impact resistance of high-speed steel which may account for the low values obtained with T-4, T-5, T-6, and T-8. A 1.1 per cent carbon type M35 high-speed steel is used for machining hardened steels and superalloys; as heat treated this steel reaches hardnesses of Rockwell C 68 to 69 with impact resistance of 6 to 8.5 ft-lb at room temperature and 16 ft-lb at 900 F. On the other hand, 1.5-3.5-1-4, a high-speed steel lean in alloy content shows unusually high impact strength at Rockwell C 63.

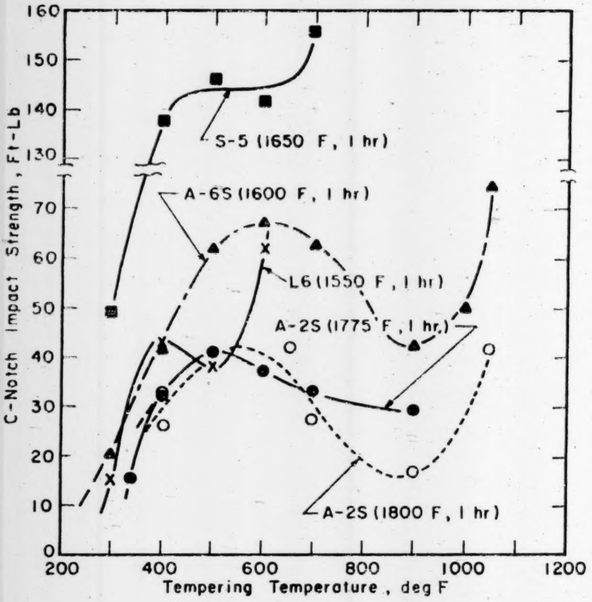

FIG. 5.—C-Notch Charpy Impact Resistance Versus Tempering Temperature for L-6, A2-S, A-6S, and S5 Tool Steels.

Table V also illustrates the effect of variations in the conventional heat treating practices on the C-notch toughness of two high-speed steels: M-2 and 1.1C Type M-35. When these steels are underaustenitized followed by conventional tempering, the as-heat-treated hardnesses are somewhat lower; C-notch impact strength, however, is raised significantly. This toughening treatment, well known to the practical tool steel metallurgist, has been previously investigated by means of unnotched Izod impact tests (14).

It is shown in Table V that the average room temperature C-notch impact resistance of M-2 (conventionally hardened Rockwell C 65) was raised from 16.5

TABLE IV.—SHOCK-RESISTANT STEELS.[a]

Steel	AISI Designation	Heat Treatment		Rockwell Hardness, C-Scale	C-Notch Charpy Impact Energy, ft-lb, at Indicated Test Temperature					
		Austenitized	Tempered		−100 F	Average	Room Temperature	Average	300 F	Average
14....	S-5	1650 F, 1 hr	300 F, 2 hr	62	17, 17, 17	17	50, 40, 45, 59	49	...	...
			400 F, 2 hr	60	30, 25, 32	29	135, 140	138	...	...
			500 F, 2 hr	59	32, 42, 42	39	138, 142, 153, 151	146	...	...
			600 F, 2 hr	58	43, 36, 36	38	137, 125, 147, 145, 160	143	...	...
			700 F, 2 hr	57	33, 39, 34	35	153, 162, 163, 144	156	...	...
15....	0.45C-Mn-Si-Cr-V-Mo Steel	1700 F, 1 hr	550 F, 2 hr	57	176, 164, 153	164	175, 188, 161	175	165, 169, 157	164
16....	L-2	1650 F, 1 hr	300 F, 2 hr	60	59, 25, 37	40	155, 124, 111	130	...	...
			400 F, 2 hr	57	129, 128, 100	119	188, 174, 94, 107, 174	146	...	...
			500 F, 2 hr	55	92, 110, 114	105	159, 125, 150, 146, 160	148	...	...

[a] All steels were oil quenched from the austenitizing temperature.

TABLE V.—MOLYBDENUM-BASE HIGH-SPEED STEELS.[a]

Steel	AISI Designation	Heat Treatment		Rockwell Hardness, C-Scale	C-Notch Charpy Impact Value, ft-lb, at Indicated Test Temperature			
		Austenitized	Tempered		76 F Tests	Average	900 F Tests	Average
17......	M-2	2225 F, 10 min[b]	1050 F, 2 + 2 hr	64	18 tests (18–23)	20	32, 33, 38, 38	35
17A.....	M-2	2225 F, 4 min	1050 F, 2 + 2 hr	65	28 tests (11–19)	16	34, 28, 31	31
17B.....	M-2	2225 F, 4 min	1050 F, 2 + 2 hr	65	12, 17, 17, 18	16	...	...
17C.....	M-2	2225 F, 4 min	1050 F, 2 + 2 hr	65	19, 12, 18, 13	15.5	...	...
17D....	M-2	2225 F, 4 min	1050 F, 2 + 2 hr	65	17, 16, 19, 17, 18	17.5	...	...
17E.....	M-2	2225 F, 10 min[b]	1000 F, 2 hr	66	17, 16, 16, 17	16.5	29, 38, 15, 15	24
17E.....	M-2	2225 F, 10 min[b]	1100 F, 2 hr	63	20, 21, 18, 23	19.5	34, 35, 27, 33	32
17E.....	M-2	2275 F, 10 min[b]	1050 F, 2 hr	63.5	5, 5, 5	5	10, 8, 10, 8	9
17E.....	M-2	2175 F, 10 min[b]	1050 F, 2 hr	62.5	19, 21, 20, 19	20	34, 34, 27, 28	30
17E.....	M-2	2075 F, 10 min[b]	1050 F, 2 hr	60.0	33, 30, 30	31	40, 40, 45, 38	41
18......	M-2S	2225 F, 4 min	1050 F, 2 + 2 hr	64	17, 14, 18, 14, 16	16	22, 20, 20, 20, 16	20
18A.....	M-2S	2225 F, 4 min	1050 F, 2 + 2 hr	64	14, 19, 17, 18, 16	17	30, 28, 30, 30, 23	28
19......	1.0C M-2S	2225 F, 4 min	1050 F, 2 + 2 hr	66	10, 10, 11	10	24, 22, 20, 20, 12	20
20......	1.0C M-2	2225 F, 4 min	1050 F, 2 + 2 hr	66.5	10, 10, 10	10	10, 16, 11, 16, 16	14
21......	M3S-1	2225 F, 1 min	1050 F, 2 + 2 hr	65	13, 12, 12, 10	12	17, 16, 15	16
22......	M3S-2	2225 F, 1 min	1050 F, 2 + 2 hr	65	10, 8, 10, 10	9.5	...	...
23......	M-7	2200 F, 4 min	1050 F, 2 + 2 hr	65	13, 13, 14, 14, 14	13.5	21, 19, 21, 20, 20	20
23A.....	M-7	2200 F, 4 min	1050 F, 2 + 2 hr	65	14, 14, 13, 13, 14	13.5	22, 20, 22, 20, 20	21
24......	High C	2100 F, 1 min	1025 F, 2 + 2 + 2 hr	66.7	12, 12, 12	12	...	...
	Type M-35	2150 F, 1 min	1025 F, 2 + 2 + 2 hr	67.9	7, 8, 8	8	...	...
		2175 F, 1 min	1025 F, 2 + 2 + 1000 F, 2 hr	67.5/68	9, 8, 8	8.5	...	...
		2200 F, 1 min	1025 F, 2 + 2 hr	68.4	7, 5, 6, 8	7	...	...
		2200 F, 1 min	1025 F, 2 + 2 + 2 hr	68.0	7, 7, 7	7	...	...
		2200 F, 1 min	1075 F, 2 + 2 + 2 hr	66.0	8, 8, 10	8.5	...	...
24A.....	High C	2215 F, 4 min	1050 F, 2 + 2 hr	69.0	12 tests (5–7)	6	16, 16, 16	16
24......	Type M-35	2225 F, 1 min	1025 F, 2 + 2 + 2 hr	68.6	4, 4, 4	4	...	...
25......	1.5-3.5-1-4	2050 F, 30 min	1000 F, 2 + 2 hr	64	14, 20, 23	19	62, 64, 75, 73, 72, 65	68
		2050 F, 30 min	1050 F, 2 + 2 hr	63	35, 47, 32, 34	37	...	...

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[a] All steels were oil quenched from the austenitizing temperature.

[b] Specimens heated in an air muffle furnace. All others were austenitized in salt bath.

TABLE VI.—TUNGSTEN-BASE HIGH-SPEED STEELS.

Steel	AISI Designation	Rockwell Hardness, C-Scale	Heat Treatment Austenitized[a]	C-Notch Charpy Impact Value, ft-lb at Indicated Test Temperature			
				Room Temperature	Average	900 F	Average
26	T-1	65	2350 F	24 Tests (Range 10–15)	12	16, 18, 16, 18, 19	17
26A	T-1	65	2350 F	8, 8, 10, 10, 12, 11, 11, 12	10.5	14, 15, 18	16
27	T-4	65	2375 F	6, 6, 6	6	13, 14, 12	13
28	T-5	65	2375 F	4, 4, 4	4	8, 9, 9	9
29	T-6	65	2375 F	3, 3, 3	3	5, 6, 5	5
30	T-8	66	2350 F	3, 4, 4	4	8, 8, 7	8
31	T-9	66	2300 F	9, 8, 9	9	14, 11, 12	12
Non-Iron Base Cutting Tool Materials							
Cobalt Base Alloy[b]	...	63	As-Cast	1, 1, 1	1	...	...
Cobalt Base Alloy[c] Cast in a Graphite Mold	...	63	As-Cast	1, 1, 2	1	...	...
Tungsten Carbide	6 per cent Cobalt Binder	91 RA[d]	...	2, 2, 2	2	2, 2, 2	2
Tungsten Carbide	12 per cent Cobalt Binder	89 RA[d]	...	2, 2, 2	2	2, 2, 2	2

[a] Austenitized 5 min in air; oil quenched and tempered at 1050 F for 2 + 2 hr.

[b] 2.0 per cent carbon, 4.0 per cent vanadium, 3.5 per cent nickel, 30 per cent chromium, 18.5 per cent tungsten, balance cobalt, 2.5 (max) iron.

[c] 2.25 per cent carbon, 33 per cent chromium, 18 per cent tungsten, 2 max iron.

[d] Rockwell A hardness scale.

TABLE VII.—TRANSVERSE AND LONGITUDINAL C-NOTCH IMPACT TOUGHNESS OF SPECIMENS MACHINED FROM LARGE HOT-WORK STEEL BARS.

Steel	Heat Treatment	Rockwell Hardness, C-Scale	Charpy C-Notch Impact, ft-lb			
			Specimens Aligned Longitudinally	Average, ft-lb	Specimens Aligned Transversely	Average, ft-lb
Specimens Cut from Material at the Extreme Bar Ends						
32	1850 F, 1 hr, air cooled, tempered 1025 F 2 + 2 hr	54	27, 20 30, 44	30	18, 10, 16, 16	15
33	1900 F, 1 hr, air cooled, tempered 1025 F 2 + 2 hr	55	52, 50, 62, 59	56	38, 28, 22, 26	28
34	1900 F, 1 hr, air cooled, tempered 1050 F 2 + 2 hr	55/56	144, 106, 146, 114	128	46, 42, 45, 40	43
Specimens Machined Close to the Center of the Bar Lengths						
32	1850 F, 1 hr, air cooled, tempered 1015 F 2 + 2 hr	54	21, 20, 20, 16	20	12, 15, 10, 9	12
33	1900 F, 1 hr, air cooled, tempered 1015 F 2 + 2 hr	55	35, 40, 18, 36	32	6, 19, 11, 9	11
34	1900 F, 1 hr, air cooled, tempered 1050 F	55/55.5	115, 140, 138	131	26, 27, 28, 39	30

ft-lb to 31 ft-lb when the steel was quenched from 2075 F and double tempered at 1050 F to Rockwell C 60. Similarly, the C-notch impact strength of 1.1C type M-35 could be increased from an average of 7 to 12 ft-lb by lowering the austenitizing temperature 100 F (from 2200 to 2100 F) followed by 3 tempers at 1025 F. In this latter comparison the specimen hardness differed about 2 Rockwell C points, that is, 68.5 to 66.7. It will be noted in passing that conventional austenitizing temperatures and higher temperature tempers do not produce equivalent toughening of the steel structure at the same specimen hardnesses.

When the austenitizing temperature is raised above the accepted range, the C-notch impact resistance tends to take a sharp drop, that is, M-2 specimens austenitized at 2275 F absorb only 5 ft-lb impact energy; 1.1C type M-35 steel austenitized at 2225 F absorbs 4 ft-lb. In either steel, the difference between the austenitizing temperature and the solidus temperature was narrowed to 20 to 25 F. Several subtle changes take place in the fine structure of overheated high-speed steel that contribute to its progressive embrittlement—grain coarsening, carbide migration to the grain boundaries, carbide fusion, and ultimately the formation of eutectic upon cooling of incipient liquid pools.

Impact test data for cutting-tool materials other than high-speed steels are shown in Table VI: namely, two grades of sintered tungsten carbide and two cast cobalt-base alloys.

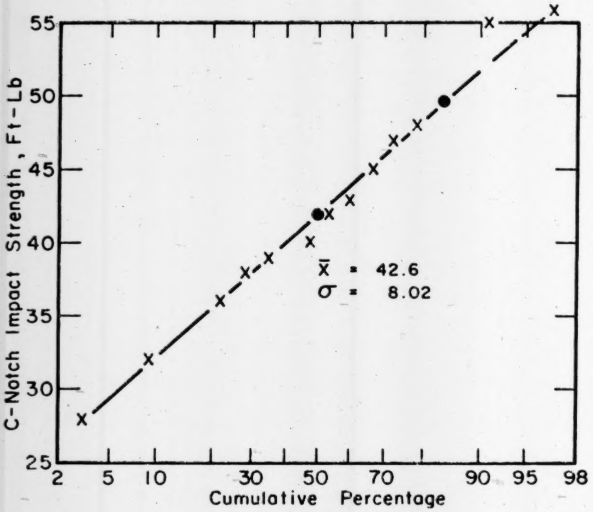

FIG. 6.—Distribution of C-Notch Impact Test Data for A-6S on Arithmetic Probability Paper. Austenitized at 1600 F, air cooled, tempered at 400 F for 2 hr; Rockwell C 59.

As would be expected, the room-temperature C-notch impact strength of the pressed-and-sintered products and the cast alloys is low and does not increase at 900 F.

Table VII illustrates the use of C-notch impact test data in comparing the longitudinal and transverse toughness of three hot-work steels forged into bars 7 by $3\frac{1}{2}$ in. by 72-in. length. These steels were selected to show the possible overlap of C-notch impact test results into a toughness range where Charpy V-notch data are equally valid, that is, the longitudinal C-notch impact specimens from both end and center locations of steel 34 broke with about 130 ft-lb absorbed energy. This corresponds to about 10 ft-lb Charpy V-notch energy absorption (Fig. 3). On the other hand, the transverse specimens of all three steels showed average C-notch impact resistances of only 11 to 43 ft-lb; at this toughness level the conventionally V-notch test would produce very low values and, therefore, would be incapable of ranking the impact toughness of the steels.

DISCUSSION

Statistical Analysis of the Data:

In assessing the data obtained with this new test method, three questions can be posed:

1. What is the statistical significance of data obtained with material from one heat of steel?
2. How reproducible are the data from heat to heat of one specific steel grade?
3. How are test results influenced by differences in specimen fabrication in different machine shops and testing variables in three laboratories?

To answer the first question, impact test data for selected steels were plotted on probability paper. In Fig. 6, 16 impact data of A-6S from Table III are plotted against cumulative percentages of sample size. The selected heat treatment of the test specimens was as follows: austenitized at 1600 F for 1 hr, air cooled, and tempered at 400 F for 2 hr. The data are arranged in order of increasing magnitude and a cumulative percentage is assigned to each datum point (Tables VIII and IX) (24).

The best straight line drawn through the data points indicates that the distribution of the test results is essentially normal. The intercept of the ordinate and the 50 per cent abscissa represents the arithmetic mean $\bar{X}$ of the impact data. The standard deviation σ has the significance that on the average 68.27 per cent of the data are included by $\bar{X} \pm \sigma$. The standard deviation was computed:

$$\sigma = \sqrt{\frac{\Sigma(X - \bar{X})^2}{n - 1}}$$

where X is the individual impact strength value and n is the number of tests made.

TABLE VIII.—ARRANGEMENT OF A-6S DATA FOR PLOTTING ON PROBABILITY PAPER.

Impact Strength, ft-lb	Cumulative Number of Observations	Cumulative Percentage Corresponding to Sample Size 16 (from Table IX)[a]
28	1	3.1
34	2	9.4
36	4	21.9
38	5	28.1
39	6	34.4
40	8	46.9
42	9	53.1
43	10	59.4
45	11	65.6
47	12	71.9
48	13	78.1
55	15	90.6
56	16	96.9

[a] The data are plotted in Fig. 6.

Figures 7 to 9 show the statistical distribution of selected C-notch impact test data of A2 sulfurized, D-2 sulfurized, and L6 for given heat treatments and hardnesses. The arithmetic means and standard deviations of these steels and of A-6S, S-5 are listed in Table X. One will observe in this table that the scatter of test data increases as the tempering temperature of L6 is raised from 300 to 600 F. Thus in Fig. 9 the sigma of the distribution curves increases with increasing tempering temperature and ranges from 3.07 ft-lb (Rockwell C 63) to 19.1 ft-lb (Rockwell C 56). The trend of increasing sigma is maintained even though the average impact strength at

TABLE IX.—CUMULATIVE PERCENTAGES CORRESPONDING TO DIFFERENT SAMPLE SIZES.

Observation Number	5	6	7	8	9	10	11	12	13	14	15	16
1	10	8.4	7.2	6.2	5.6	5	4.5	4.2	3.8	3.6	3.3	3.1
2	30	25.0	21.4	18.8	16.6	15	13.6	12.5	11.5	10.7	10.0	9.4
3	50	41.6	35.6	31.4	27.6	25	22.7	20.8	19.2	17.8	16.7	15.6
4	70	58.2	49.8	44.0	38.6	35	31.8	29.2	26.9	25.0	23.3	21.9
5	90	74.8	64.0	56.6	49.6	45	40.9	37.5	34.6	32.1	30.0	28.1
6	...	91.4	78.2	69.2	60.6	55	50.0	45.8	42.3	39.2	36.7	34.4
7	...	...	92.4	81.8	71.6	65	59.1	54.2	50.0	46.4	43.3	40.6
8	...	...	...	94.4	82.6	75	68.2	62.5	57.7	53.5	50.5	46.9
9	...	...	...	...	93.6	85	77.3	70.8	65.4	60.7	56.7	53.1
10	...	...	...	...	...	95	86.4	79.2	73.1	67.8	63.3	59.4
11	...	...	...	...	...	...	95.5	87.5	80.8	75.0	70.0	65.6
12	...	...	...	...	...	...	...	95.8	88.5	82.1	76.7	71.9
13	...	...	...	...	...	...	...	...	96.2	89.2	83.3	78.1
14	...	...	...	...	...	...	...	...	...	96.4	90.9	84.4
15	...	...	...	...	...	...	...	...	...	...	96.7	90.6
16	...	...	...	...	...	...	...	...	...	...	...	96.9

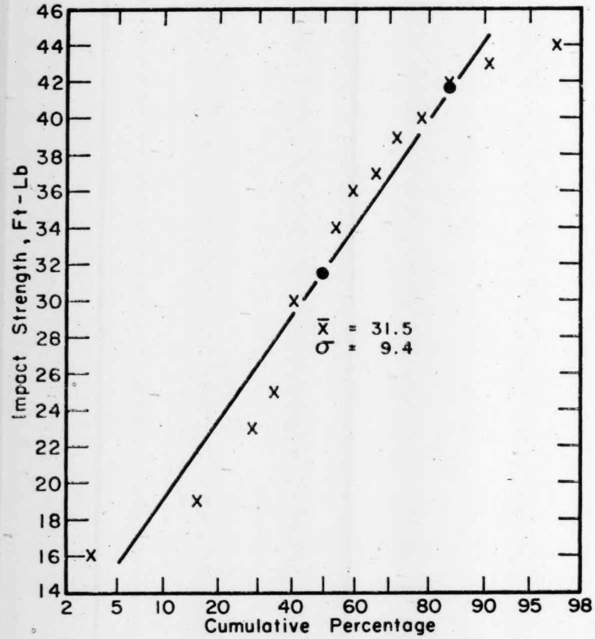

FIG. 7.—Distribution of C-Notch Impact Test Data on Arithmetic Probability Paper. A-2S austenitized at 1775 F, air cooled and tempered 2 hr at 400 F; Rockwell C 61.

Rockwell C 58 is lower than that at Rockwell C 60. The error function about the mean impact value can be also expressed as a percentage of the mean, $[\sigma_x/\bar{X} \times 100]$, which is the coefficients of variation. These new functions show a similar rise with increasing tempering temperatures, that is, decreasing material hardnesses (see last column in Table X). However, with very tough

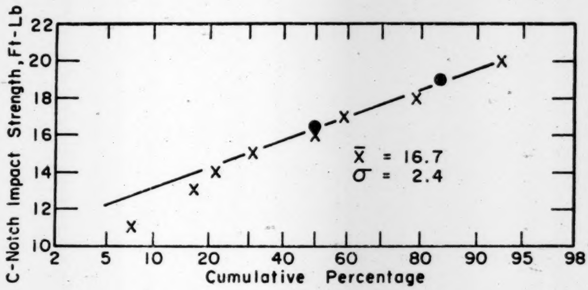

FIG. 8.—Distribution of C-Notch Impact Test Data on Arithmetic Probability Paper. D-2S austenitized at 1850 F, air cooled and tempered 3 hr at 250 F; Rockwell C 64.

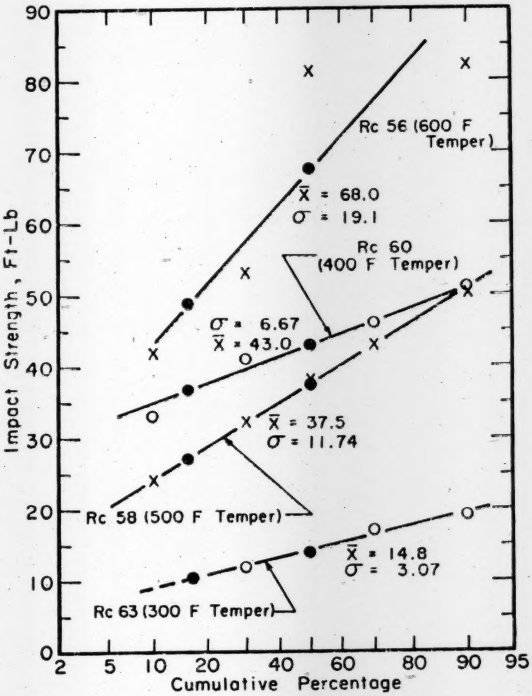

FIG. 9.—Effect of As-Tempered Hardness of L6 on the Standard Deviation from the Mean Impact Strength.

Austenitized at 1550 F for 1 hr, tempered at 300 to 600 F for 2 hr.

steels, that is, S-5 tempered at 600 F, the impact resistance appears to rise faster than the coefficient of variation, so that $\bar{\sigma}_x/\bar{X}$ is again lower. It is reasonably certain that larger samples of the different universes would yield corresponding results.

Figure 10 shows the statistical evaluation of 53 C-notch impact tests representing three heats of M-2 high-speed steel. All specimens had received a uniform heat treatment as follows: 2225 F, 4 min in salt, quenched in oil to room temperature, and tempered at 1050 F, 2 + 2 hr. The arithmetical mean of the impact data is 16.37 ft-lb with a $\bar{\sigma}_x$ of 3.08, a $2\bar{\sigma}_x$ of 6.16 and a $3\bar{\sigma}_x$ of 9.24 ft-lb representing 68.2, 95.5, and 99.7 per cent of the area under the normal distribution curve. These values are compared in Table XI with the average impact resistance and standard deviation of a second specimen population taken from three different heats of commercial M-2 steel. The arithmetic mean of 16.40 ft-lb and $3\bar{\sigma}_x$ = 6.44 ft-lb agrees well with the results of the first test series. Table XI also compares the coefficient of variation of the above tests with results reported in the literature for the unnotched Izod impact test (reference (**14**) and discussion of this paper). Using heat treatments similar to those of the present investigation, Grobe and Roberts achieved low scatter in duplicate unnotched impact tests of 2 single M-2 heats. The corresponding test results reported in discussions of this paper show coefficients of variation intermediate between those of the C-notch tests and the unnotched Izod tests of the original investigation.

TABLE X.—STATISTICAL EVALUATION OF C-NOTCH CHARPY IMPACT TESTS DATA OF L-6 (AT FOUR HARDNESSES), A6-S, A2-S, D2-S, AND S-5.

AISI Designation	Heat Treatment		Rockwell Hardness, C-Scale	Number of Tests	Room Temperature C-Notch Impact Strength, ft-lb		
	Austenitized	Tempered			Arithmetic Mean, $\bar{X}$	Standard Deviation, σ	Coefficient of Variation, $\bar{\sigma}_x/\bar{X}$ × 100 per cent
L-6	1550 F, 1 hr	300 F, 2 hr	63	5	14.8[a]	3.07	20.7
		400 F, 2 hr	60	5	43.0[a]	6.67	15.5
		500 F, 2 hr	58	5	37.5[a]	11.74	31.3
		600 F, 2 hr	56	5	68.0[a]	19.1	28.1
A6-S	1600 F, 1 hr	400 F, 2 hr	59	16	42.6[b]	8.0	18.2
A2-S	1775 F, 1 hr	400 F, 2 hr	61	16	31.5[b]	9.4	29.8
D2-S	1850 F, 1 hr	250 F, 2 hr	64	17	16.7[b]	2.4	18.5
S-5	1650 F, 1 hr	600 F, 2 hr	58	5	143[c]	13.0	9.1

[a] Data from Table II.
[b] Data from Table III.
[c] Data from Table IV.

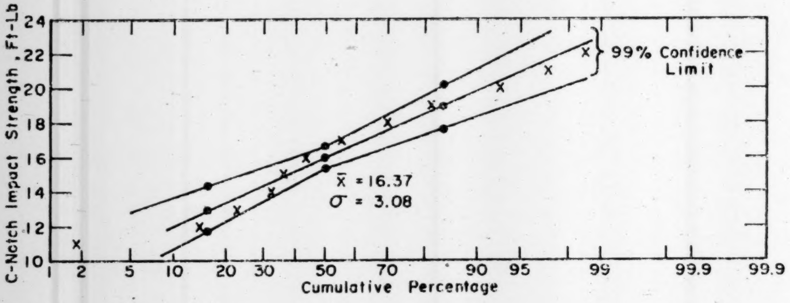

FIG. 10.—Statistical Evaluation of 53 C-Notch Charpy Impact Tests Representing Three Heats of Rex M-2 (Steels Nos. 17, 17A, and 17B).

Austenitized at 2225 F, 4 min oil quenched and tempered at 1050 F for 2 + 2 hr.

The coefficients of variation of the

TABLE XI.—STATISTICAL ANALYSIS OF CHARPY C-NOTCH AND UNNOTCHED IZOD IMPACT TEST RESULTS FOR M-2 HIGH SPEED STEEL.

C-Notch Impact Strength, 2225 F, 4 min salt, oil quenched and tempered 1050 F, 2 + 2 hr Specimen 0.394 sq in., ½-in. notch					Unnotched Izod Impact Strength							
					2225 F, semimuffle furnace ½ min oil quenched, 1050 F, 2 × 2½ hr Specimen 0.394 sq in.[a]				2250 F, salt, oil quenched and tempered 1050 F, 2½ + 2½ hr Specimen 0.375 sq in.[b]			
Present Investigation	Number of Specimens	Average Impact Strength, $\bar{X}$, ft-lb	$3\bar{\sigma}_x$, ft-lb	$3\bar{\sigma}_x \times 100/\bar{X}$, per cent	Reference (14)	Average Impact Strength,[a] $\bar{X}$, ft-lb	$3\bar{\sigma}_x$, ft-lb	$3\bar{\sigma}_x \times 100/\bar{X}$, per cent	Discussion of Reference (14)	Average Impact Strength,[b] $\bar{X}$, ft-lb	$3\bar{\sigma}_x$, ft-lb	$3\bar{\sigma}_x \times 100/\bar{X}$, per cent
Heats 17, 17A, 17E	53	16.37	9.18	56	First Heat Lot	48.7	2.8	5.8	First Heat Lot	41.0	6.8	16.6
Heats 17B, 17C, 17D	13	16.4	6.44	39	Second Heat Lot	47.7	5.8	12	Second Heat Lot	35.0	9.6	27.4

[a] Duplicate tests.

[b] Ten tests per heat lot.

TABLE XII.—COMPARISON OF C-NOTCH IMPACT STRENGTH OF M-2, T-1, AND TWO OTHER STEELS FABRICATED BY THREE DIFFERENT MACHINE SHOPS AND TESTED AT ONE LABORATORY.

High-Speed Tool Steel[a]	Machine Shop	C-Notch Charpy Impact Energy, ft-lb	Average, ft-lb
Composition X	A	13, 14, 14, 15	14
	B	15, 16, 12, 8	13
	C	16, 14, 15, 10 ,11, 11, 12	13
M-2(Steel No. 17 A)	A	12, 17, 17, 18	16
	B	19, 12, 18, 13	15.5
	C	12, 17, 18, 16 ,18 ,17, 16, 18	16.5
Composition Y	A	13, 10, 8, 11	10.5
	B	12, 12, 12, 12	12
	C	8, 8, 10, 8, 9, 11, 8	9
T-1 (Steel No. 26)	A	12, 12, 12, 13	12
	B	12, 12, 12, 12	12
	C	14, 12, 13, 13, 12, 15, 15, 15, 12	13.5

[a] M-2 was austenitized at 2225 F and T-1 at 2350 F. All specimens were oil quenched and tempered at 1050 F for 2 + 2 hr.

C-notch impact test obtained from two groups of three M-2 heats each, establish a normal expectancy of impact toughness variations from heat to heat of the steel.

A statistical evaluation was made of unnotched Charpy impact test data for high hardness steels (**15**). These investigators determined the need for specimen lot sizes of 30 to 40 for each condition of the steels to achieve a maximum ±10 per cent error at the 95 per cent confidence limit for the mean impact values.

It was now of interest to check the effect of the following variables on the reproducibility of impact test results: (1) fabrication of specimens by different shops and (2) impact testing by different laboratories.

Table XII compares the C-notch impact strength of four high-speed steels when the test specimens were finish

TABLE XIII.—ANALYSIS OF VARIANCE OF C-NOTCH IMPACT TEST RESULTS FOR COMPOSITION X. (EVALUATION OF DIFFERENCES IN GRINDING PRACTICE IN THREE MACHINE SHOPS).

Test Number	C-Notch Impact Test Results from Table XII Specimen Finish Ground in Shop				A	B	C	Σ
	A	B	C					
1	13	15	16	Sum of Individual Observations	56	51	89	196
2	14	16	14					
3	14	12	15					
4	15	8	10	Sum of Squares (SS)	786	689	1,163	2,638
5	...	...	11					
6	...	...	11					
7	...	...	12					
Sum of Individual Observations	56	51	89	Observations	4	4	7	$N = 15$

$$\text{Total SS} = 2{,}638 - \frac{(196)^2}{15} = 77$$

$$\begin{aligned} \text{Shop SS} &= \frac{(56)^2}{4} + \frac{(51)^2}{4} + \frac{(89)^2}{7} - \frac{(196)^2}{15} \\ &= 784 + 650.2 + 1{,}131.7 - 2561.0 \\ &= 4.9 \end{aligned}$$

Source of Variation	Sum of Squares (SS)	Degrees of Freedom (df)	Mean Square = SS/df	Ratio of Among-Shops Mean Square to Error Mean Square, F
Total	77.0	$(N - 1) = 14$	5.5	...
Among Shops	−4.9	(No. of Shops—1) −2	2.45	0.408
Error	72.1	12	6.01	...

If one consults an F table with 2 and 12 degrees of freedom, the F necessary to claim a significant difference in impact values between shops at the 5 per cent level of significance, $F_{0.05} = 3.89$. The F value obtained in the above calculation is substantially less than $F_{0.05}$; therefore, it must be concluded that there is no statistical difference between average C-notch impact test results of specimens ground in machine shops A, B, and C.

ground by three different machine shops. Test specimens of the same steel were simultaneously heat treated in a salt bath. The three shops then ground individually ½-in. radii in specimens from each of the four groups of M-2, T-1, and compositions X and Y, using normal care in the grinding operations. The specimens were tested on a Riehle impact testing machine.

An analysis of variance (25) shows that the average impact resistance of the steels is not significantly affected by shop fabrication variables of the specimens. Table XIII shows the analysis of variance of the three groups of steel X as an illustration of the method of calculations used.

The four high-speed steels listed in Table XII were also used to check the reproducibility of test results among testing machines in different laboratories. In this test, specimens from each grade were fabricated by the same shop and heat treated simultaneously by conventional practice. Impact tests were conducted at Crucible Steel Co.'s Central and Midland Research Laboratories, respectively, and at Watertown Arsenal. The impact testing machines at the Crucible Laboratories had been certified by Watertown Arsenal shortly before the tests were made.

Table XIV, listing the impact data obtained for each steel on three testing machines, shows a small but consistent spread in average impact strength reported by the three laboratories.

TABLE XIV.—COMPARISON OF C-NOTCH IMPACT DATA OF M-2, T-1 AND 2 OTHER STEELS OBTAINED FROM TESTS AT THREE DIFFERENT LABORATORIES.

High-Speed Tool Steel	Laboratory Conducting Tests[a]	C-Notch Charpy Impact Energy, ft-lb	Average, ft-lb
M-2	A	17, 16, 19, 12, 17, 18, 16, 18, 17, 16, 18	16.7
	B	16, 14, 18, 14	15.5
	C	11, 15, 14, 14	13.5
Composition X	A	13, 14, 14, 15, 15, 12, 8	13.4
	B	12, 12, 12	12.0
	C	10, 11, 11, 12	11.0
Composition Y	A	13, 10, 8, 11, 12, 12, 12, 12	11.2
	B	8, 8, 10	8.6
	C	8, 9, 11, 8	9.0
T-1	A	12, 12, 12, 13, 12, 12, 12, 12	12.1
	B	11, 11, 10, 12	11.0
	C	11, 10, 11, 10	10.5

[a] A = Central Research Laboratory of Crucible Steel Co. of America (Riehle testing machine).
B = Midland Research Laboratory of Crucible Steel Co. of America (Sontag impact testing machine).
C = Watertown Arsenal (Mouton Charpy testing machine).

From the limited data, however, it appears that only M-2 and composition Y show a statistically significant effect of the testing machines on impact test results.

SUMMARY AND CONCLUSIONS

For a ready comparison, Fig. 11 is a plot of C-notch impact strength versus Rockwell C hardness of some conventionally hardened tool steels. It confirms the well known fact that high carbon content generally is inimical to high impact resistance. Thus, the tool steels with 0.90 to 2.20 per cent carbon (O-1, A-2 sulfurized, D-2 sulfurized, L-7, and D-4), when heat treated to Rockwell C 58 to 62, yielded C-notch impact strength of about 40 ft-lb or less. Steels with 0.70 to 0.75 per cent carbon (A-6S and L-6 resulfurized) show intermediate C-notch toughness of 40 to 60 ft-lb when the hardness is dropped to Rockwell C 56 to 58. Only with carbon contents in the range 0.50 to 0.60 per cent is it possible to achieve C-notch impact values of 140 to 160 ft-lb at Rockwell C 58 to 60. The findings of this investigation can be summarized as follows:

1. Charpy impact tests with a modified specimen ($\frac{1}{2}$-in. notch radius) are able to distinguish the impact toughness of tool steels heat treated to at least Rockwell C 56 and above. This test should not be used for steel compositions or hardnesses where the V-notch impact test is sufficiently selective.
2. The C-notch impact energies absorbed in testing high-hardness steels is within the range of commercial impact testing machines.

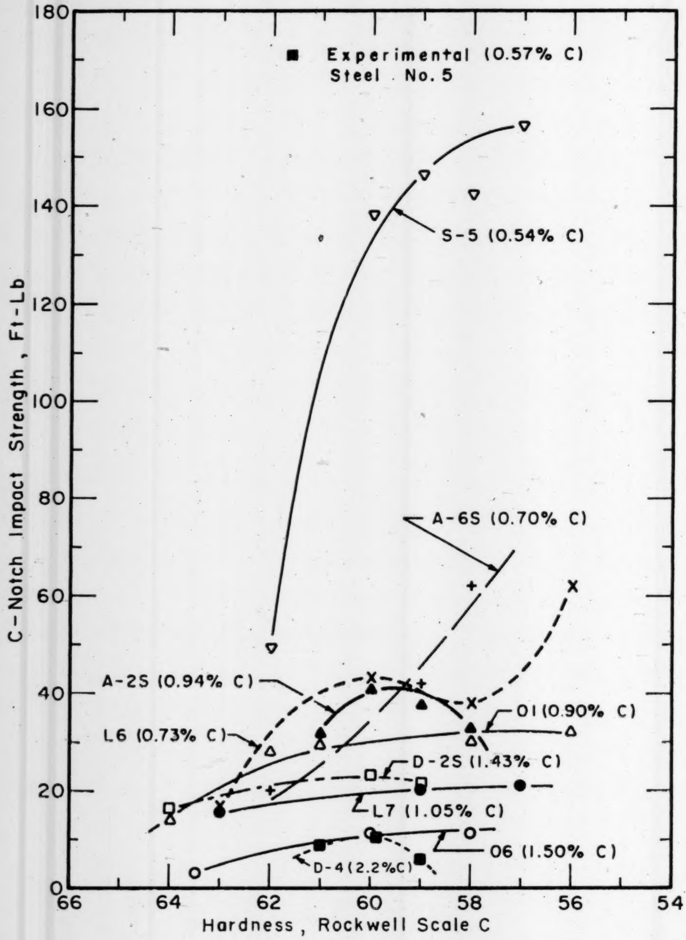

FIG. 11.—Effect of Tool Steels Carbon Content and Hardness Level on Attainable C-Notch Toughness.

3. C-notch impact tests can be made at room temperature and at elevated temperatures such as the temperatures reached at the cutting edges of high-speed steel tools.

4. Applied to oil hardening, air hardening, and shock resisting steels, the test brackets the impact resistance of tool steel classes and will indicate preferred heat treatments for optimum impact toughness. The test cannot predict the service life of a tool-steel part where applied stresses and loading rates are different from those found in this test or where metal fatigue must be considered.

5. The test is a valuable guide in developing alloys which achieve good toughness at very high hardnesses.

6. The test is useful in comparing directional impact strengths in large sections of nominally tough hot-work steels, when transverse toughness drops below the selective range of the conventional Charpy V-notch test.

7. With high-speed tool steels, C-notch

impact resistance relates to service experience in interrupted cutting. The elevated-temperature test data appear to be most significant for ranking the steels. Thus the molybdenum base high-speed steels (including 1.1 C—type M-35) absorb 16 to 35 ft-lb average impact energy at 900 F; the tungsten-base high-speed steels fail at 5 to 17 ft-lb; and tungsten carbides and cast cobalt-base alloys show only 1 to 2 ft-lb impact resistance. However, C-notch impact data have little or no bearing on the cutting-tool performance in continuous cutting such as in lathe turning.

8. The scatter of C-notch test data generally is normally distributed and increases when the test specimens of the same steel are tempered to lower hardnesses.

9. Impact specimens from the same heat of high-speed steel machined by different machine shops but tested in one laboratory show no significant effect of fabrication variables on average impact strength. Groups of identically treated high-speed steel specimens were also tested by three laboratories. The data showed a small but consistent spread between the average impact strength of two of the steels as obtained on different testing machines.

Acknowledgment:

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AN INVESTIGATION OF SUBSIZE CHARPY IMPACT SPECIMENS*

By N. H. Fahey[1] and E. B. Kula[1]

Synopsis

A variety of Charpy impact test specimens are used in determining the toughness of materials. The ASTM, for example, recognizes seven specimen sizes as standard; yet there has been no way to compare directly test values between different sizes or geometries.

Using AISI 4340 steel bar quenched and tempered to tensile strength levels between 138,000 and 287,000 psi, the results of the seven standard and four nonstandard specimens are compared. Tests were conducted over a range of temperatures, and comparisons were made between energy and fracture appearance. The data demonstrate the effect of size and geometry on the transitional behavior of the material investigated. Interrelations based on energy, fracture appearance, specimen size, and geometry are illustrated and discussed. Based on the assumption that the energy required to fracture subsize specimens should vary linearly with specimen width and with some function of the depth, a correlation between the energies has been established which worked satisfactorily for several steel compositions.

Thin-walled components often preclude the use of full-size Charpy specimens. This is recognized in both the ASTM and Federal Test Method procedures which recommend several specimen sizes as standard even though interrelations between test values from specimens of differing sizes or geometries have in general proved unsatisfactory. Such attempts at correlation have been unsuccessful mainly because investigators have not resolved the effect of size or geometry on the transitional behavior of materials. It has long been recognized that transition temperature changes with specimen size; this shift in transition has for the most part prevented correlation attempts based on a single test temperature unless these temperatures are above or below the transitional range of the particular material being compared.

Curll has interrelated the energy values of different size specimens fo- certain materials at -40 C, the temr perature used by the U. S. Army for most inspection tests (**1**).[2] He coupled the law of similitude with the assumption that the total energy for fracturing a Charpy specimen is made up of the energy to rupture plus the energy to deform ($E = E_r + E_d$) in order to obtain a correlation between different sizes. As the specimen size is reduced, the volume of deformation is reduced. The amount of reduction of deformed volume, however, is dependent upon the particular dimension reduced. That is, if the depth dimension is reduced, the volume of

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* The statements and opinions expressed in this article are those of the authors and do not necessarily indicate the views or policy of the U. S. Army Materiel Command.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

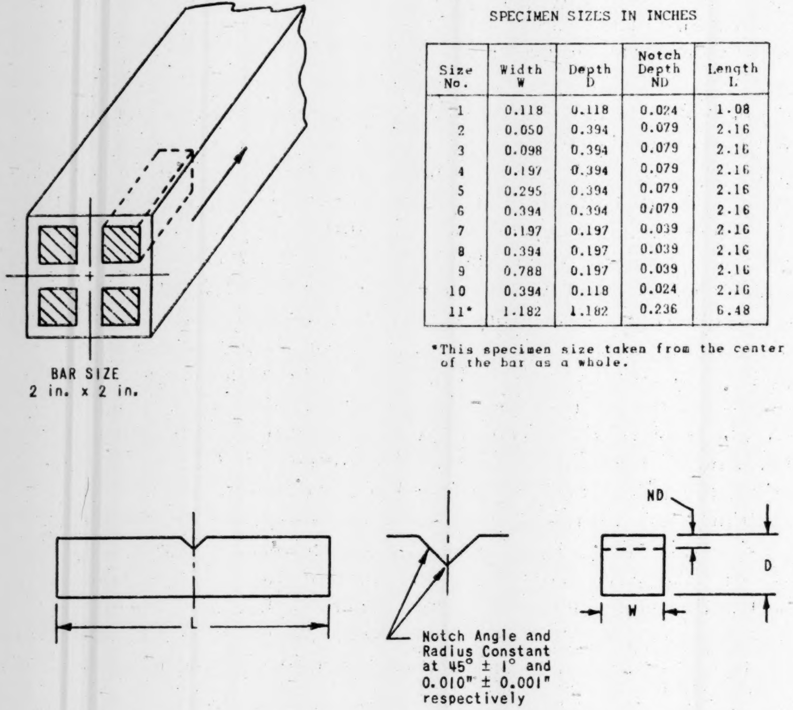
TABLE 1.—HEAT TREATMENT AND MECHANICAL PROPERTIES OF AISI 4340 STEEL.

Heat Treatment:

Specimen Identity	Temperature, deg F	Time of Treatment, hr	Cooling Medium
	1650	1½	air cool
	1600	1½	oil quench
"A" series	400	1½	oil quench
"B" series	600	1½	oil quench
"C" series	1000	1½	oil quench
"D" series	1200	1½	oil quench

Mechanical Properties:

Specimen Identity	Diameter, in.	Yield Strength, 0.1 per cent Offset, psi	Yield Strength, 0.2 per cent Offset, psi	Tensile Strength, psi	Elongation, per cent	Reduction of Area, per cent	Rockwell Hardness, C scale
A	0.357	220 000	232 500	287 000	12.9	48.6	52
B	0.357	221 000	223 000	250 000	12.9	53.7	48
C	0.357	167 500	167 000	176 800	15.7	55.9	38
D	0.357	126 000	127 000	138 800	22.9	63.0	30



SPECIMEN SIZES IN INCHES

Size No.	Width W	Depth D	Notch Depth ND	Length L
1	0.118	0.118	0.024	1.08
2	0.050	0.394	0.079	2.16
3	0.098	0.394	0.079	2.16
4	0.197	0.394	0.079	2.16
5	0.295	0.394	0.079	2.16
6	0.394	0.394	0.079	2.16
7	0.197	0.197	0.039	2.16
8	0.394	0.197	0.039	2.16
9	0.788	0.197	0.039	2.16
10	0.394	0.118	0.024	2.16
11*	1.182	1.182	0.236	6.48

*This specimen size taken from the center of the bar as a whole.

FIG. 1.—Location of Specimen Blanks from Original Bar Stock and Dimensions of Specimen Sizes Investigated.

deformation will be reduced by the square of the depth dimension; but if the width dimension is reduced, the volume of deformation will be reduced in direct proportion. Furthermore, if the material is so notch-sensitive that negligible deformation will result, then the energies of the specimens will again be directly proportional to their sizes. In this work the specimens were not geometrically similar, nor were the test conditions similar; therefore his assumptions for subsize specimens were not fully justified. By assuming the energy to rupture to be constant (differing only for ductile or brittle materials), Curll enjoyed limited success in relating the values for some materials.

Several other investigators (**2–6**) have pointed out that there are interrelations between energy and specimen appearance; some relate ductility (**2**,**3**) by measuring the deformed surfaces while others measure percentages of fibrous or crystalline fracture (**4–6**). Fahey, while investigating the feasibility of a more economical test specimen for impact testing, found that when using a concentrically notched round specimen, energy-fracture appearance interrelations were evident even for subsize specimens (**7**). As a result of these findings, this investigation was initiated to understand better the subsize behavior of Charpy specimens.

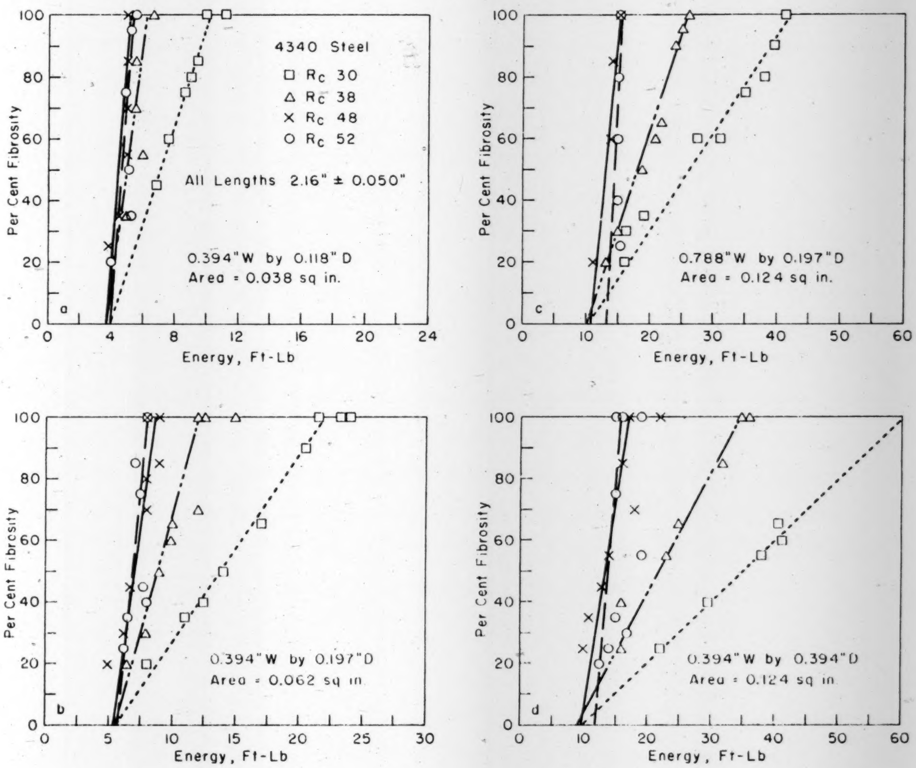


FIG. 2.—Effect of Fracture Appearance upon Energy of Varying Size Charpy Specimens of 4340 Steel at Four Strength Levels.

MATERIALS AND EXPERIMENTAL PROCEDURE

Subsize specimens were prepared by machining and grinding AISI 4340 steel bar stock quenched and tempered to tensile strength levels of 138,000 to 287,000 psi (Table 1). Whereas the material used was 2-in. square bar, four specimen blanks were cut from each length except for the largest specimen size, 1.182 in. square by 6.28 in. long. Figure 1 illustrates the locations from which the specimen blanks were cut and the sizes investigated. It should be noted that the notch angle and radius for all specimens were kept constant at 45 deg and 0.010 in. respectively and that all blanks were rough-machined to within 0.025 in. of finish specimen dimensions prior to heat treating.

With the exception of the largest size investigated, all specimens were tested in a 240 ft-lb capacity Sonntag Universal Charpy machine which was checked for accuracy in the manner outlined by Driscoll (8). The largest specimens were tested in a 2200 ft-lb capacity Mouton-Pendule machine which because of its design could not be certified with standardized specimens (8). Therefore for the largest specimen sizes, only the fracture appearance values and not the energy values were used in this investigation. To maintain the point of impact at the center of percussion of the pendulum, special anvils and shims were provided when nonstandard specimens were tested. Specimens of each size and condition were tested over a range of temperatures encompassing the ductile-to-brittle transitional range for each condition.

RESULTS AND DISCUSSION

One of the more important relations derived from the Charpy test is the linear interrelation between the energy required to fracture a specimen and the percentage of fibrous or crystalline fracture evident in the broken specimens. This can be expressed as

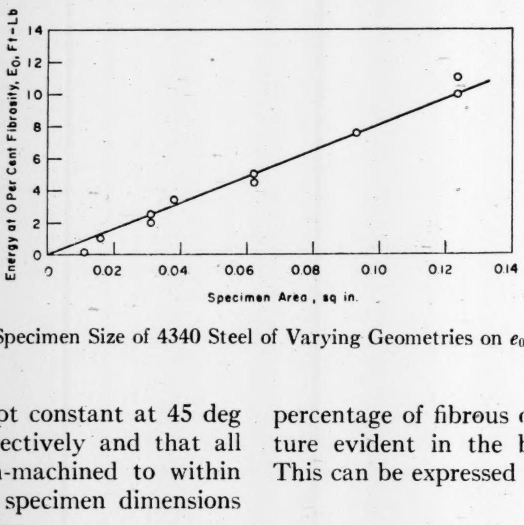


FIG. 3.—Effect of Specimen Size of 4340 Steel of Varying Geometries on e_0, Energy at Zero Per Cent Fibrosity.

$$e = e_0 + (e_{100} - e_0)\frac{F}{100} \quad \ldots\ldots (1)$$

where F is the per cent fibrosity in the fracture and e, e_0, and e_{100} are the energies at F, 0, and 100 per cent fibrosity respectively. This interrelation has been shown by many investigators for full-size square specimens and was extended (7) to include subsize concentrically notched round specimens. If such an interrelation could be found with subsize square or rectangular specimens, it would indicate a good possibility for correlating the energy values between the specimens.

In Fig. 2 the energy for fracture is plotted as a function of per cent fibrous fracture for four different sizes or geometries and four strength levels. From these curves, several observations are readily apparent. First, the linear relationship between energy and fibrosity (Eq 1) is valid for other sizes besides the standard. Second, by extrapolating each of the lines to zero per cent fibrosity, it is seen that this value, e_0, to a first approximation is independent of strength level for each of the sizes investigated. It will be noted from Figs. 2(*c*) and (*d*) that similar results were obtained from specimens of equivalent area but with differing geometries. Similar curves were constructed for each of the other specimen sizes and in each case the value at zero fibrous intercept, e_0, is independent of strength level. Other investigations encompassing a greater range of strength levels have shown that e_0 may actually vary somewhat (**5,6**). The variation is far less for e_0 than for the energy at higher fibrosity levels, however, and for the range of strength levels studied here e_0 can be assumed to be dependent only on size.

Since e_0 is the energy required to fracture a specimen of a given size and geometry with a minimum of plastic deformation, it would be expected that it should be a function of specimen size only. Figure 3 illustrates this to be so as the zero per cent fibrous energy values for all ten sizes investigated fall on one straight line. What is particularly interesting about this interrelation is that it includes data from a variety of geometries, including edge notch, face notch, and square. These findings are in agreement with the observations of Curll (**1**) who showed that if a material is so notch-sensitive that negligible deformation will result, this "energy of rupture," e_0, will be directly proportional to specimen area. He also theorized that when comparing the energy value from subsize specimens where the reduced dimension is the width alone, the volume of deformation (and thus the energy necessary to fracture the specimen) should be reduced in direct proportion.

In this investigation, energy comparisons were made at a constant percentage of fibrous fracture, where the 100 per cent fibrous level was selected. If the energy values at 100 per cent fibrosity continue to increase with increasing test

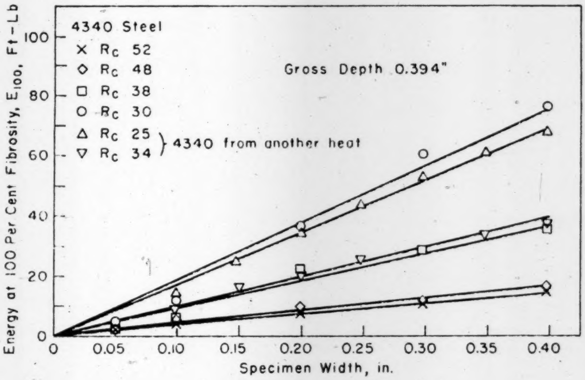


FIG. 4.—Effect of Specimen Width at Constant Depth on e_{100}, Energy at 100 Per Cent Fibrous Fracture at Six Strength Levels of 4340 Steel.

temperature, the energy value chosen is the lowest at which complete fibrous fracture persists. By plotting this value, e_{100}, for each specimen width (five sizes in all) at each strength level, a series of straight lines are obtained (Fig. 4). It should be noted that each tempering temperature (and thus each energy level) produces its own straight line. Supplementary data are included in Fig. 4 from another heat of 4340 steel with two other heat treatments used in another program. All data show very good agreement and indicate that if the specimen depth is constant, the total energy necessary for fracture at 100 per cent fibrosity, e_{100}, is then a linear function of the width.

It should now be interesting to determine if interrelations can be established by reducing either the depth alone or some combination of the depth and width. It has been shown that the total energy involved in fracturing a specimen may be expressed by Eq 1, given earlier. From Eq 1 we can assume that there are two contributions to the total energy, e_0 and $(e_{100} - e_0)\ F/100$. The term e_0 is shown in Fig. 3 to be a function of specimen area and may be referred to as the energy required to fracture a specimen of a given size and geometry in the absence of fibrous fracture and with a minimum of accompanying plastic deformation. The term $(e_{100} - e_0)\ F/100$ can be considered the energy of deformation, the extra energy arising from the plastically deformed volume associated with the fibrosity. Figure 3 has shown:

$$e_0 = K\ (\text{area}) = Kwd \ldots\ldots\ldots \tag{2}$$

where K is a constant, and w and d are the specimen width and unnotched depth.

Since:

$$E_0 = KWD \ldots\ldots\ldots\ldots \tag{3}$$

where the upper case letters refer to the full size bar, then:

$$e_0 = E_0 \frac{wd}{WD} \ldots\ldots\ldots\ldots \tag{4}$$

The energy of deformation, $(e_{100} - e_0)\ F/100$, depends on the volume deformed; therefore:

$$(e_{100} - e_0)\frac{F}{100} = K'\ (\text{volume}) = K'wdl \ldots \tag{5}$$

where l is the length of the deformed zone.

Curll assumed the length of the deformed zone was proportional to the depth, so that:

$$(e_{100} - e_0)\frac{F}{100} = K'\ wd^2 \ldots\ldots \tag{6}$$

for a full size for

$$(E_{100} - E_0)\frac{F}{100} = K'\ WD^2 \ldots\ldots \tag{7}$$

so that

$$e_{100} - e_0 = (E_{100} - E_0)\frac{wd^2}{WD^2} \ldots\ldots \tag{8}$$

Combining Eqs 1, 4, and 8, one obtains:

$$e = E_0 \frac{wd}{WD} + (E_{100} - E_0)\frac{wd^2}{WD^2}\frac{F}{100} \ldots \tag{9}$$

This equation is essentially the same as Curll's (1) except that instead of E_0 he used a constant, K, which varied for brittle or ductile materials.

Using the data for the standard size bars, energy values were calculated for the subsize bars at the 100 per cent fibrous level from Eq 9. In most cases the calculated values were found to be low. A thorough examination of the data showed that the assumption that the length of the deformed zone varies linearly with the specimen depth is not valid and that a more complex relationship is called for. When the gross depth was 0.394 in., the energy was simply:

$$e_{100} = E_{100}\frac{w}{W} \ldots\ldots\ldots\ldots \tag{10}$$

TABLE 2.—COMPARISON OF CALCULATED AND ACTUAL ENERGY VALUES FOR STANDARD AND SUBSIZE SPECIMENS.

NOTE.—For gross depth of 0.394 in., $e_{100} = E_{100} \frac{w}{W}$.

For gross depth of 0.118 and 0.197 in., $e_{100} = E_0 \frac{wd}{WD} + 1.35 \, (E_{100} = E_0) \, \frac{w}{W} \left(\frac{d}{D}\right)^2$.

In above equations, depth under notch (not gross depth) is used.

Material	Size Width by Gross Depth, in.	Calculated Energy, ft-lb	Actual Energy, ft-lb
AISI 4340, heat treated "A," E_{100} = 17.0 ft-lb	0.118 × 0.118	1.4	1.3
	0.050 × 0.394	2.5	2.5
	0.098 × 0.394	4.5	4.5
	0.197 × 0.394	9.0	8.0
	0.295 × 0.394	13.0	12.5
	0.394 × 0.394	17.0	17.0
	0.197 × 0.197	3.7	4.0
	0.394 × 0.197	7.4	7.5
	0.788 × 0.197	14.7	15.0
	0.394 × 0.118	4.7	5.0
AISI 4340, heat treated "B," E_{100} = 18.0 ft-lb	0.118 × 0.118	1.4	1.2
	0.050 × 0.394	2.5	2.6
	0.098 × 0.394	4.5	4.5
	0.197 × 0.394	10.0	10.5
	0.295 × 0.394	13.0	12.0
	0.394 × 0.394	18.0	18.0
	0.197 × 0.197	3.9	4.2
	0.394 × 0.197	7.7	8.5
	0.788 × 0.197	15.4	14.5
	0.394 × 0.118	4.8	5.0
AISI 4340, heat treated "C", E_{100} = 36.0 ft-lb	0.118 × 0.118	2.0	1.6
	0.050 × 0.394	5.0	3.0
	0.098 × 0.394	9.5	6.0
	0.197 × 0.394	19.0	20.0
	0.295 × 0.394	28.0	29.0
	0.394 × 0.394	36.0	36.0
	0.197 × 0.197	6.9	7.0
	0.394 × 0.197	13.8	13.0
	0.788 × 0.197	27.5	27.0
	0.394 × 0.118	7.1	7.0
AISI 4340, heat treated "D," E_{100} = 77.0 ft-lb	0.118 × 0.118	3.5	3.0
	0.050 × 0.394	10.0	5.0
	0.098 × 0.394	18.0	11.0
	0.197 × 0.394	37.0	40.0
	0.275 × 0.394	57.0	61.0
	0.394 × 0.394	77.0	78.0
	0.197 × 0.197	13.7	11.0
	0.394 × 0.197	27.6	24.0
	0.788 × 0.197	55.0	46.0
	0.394 × 0.118	12.2	10.5
DATA FROM CURLL, REFERENCE (1)			
Table I, E_{100} = 39.5 ft-lb	0.394 × 0.197	15.1	15.2
	0.394 × 0.118	7.9	8.2
	0.197 × 0.197	7.1	8.1
Table II, E_{100} = 29.5 ft-lb	0.394 × 0.197	11.9	10.5 to 12.8
	0.394 × 0.118	6.8	6.8
	0.197 × 0.197	6.9	6.5
Table III, E_{100} = 26.0 ft-lb	0.394 × 0.197	10.4	9.8
	0.197 × 0.197	5.2	6.1
Table IV, E_{100} = 36.0 ft-lb	0.394 × 0.197	13.8	13.0
	0.394 × 0.118	7.3	6.8
	0.197 × 0.197	6.9	6.5

TABLE 2.—*Concluded*

Material	Size Width by Gross Depth, in.	Calculated Energy, ft-lb	Actual Energy, ft-lb
Table V, E_{100} = 33.0 ft-lb	0.394 × 0.197	12.8	13.6
	0.394 × 0.118	6.9	7.9
	0.197 × 0.197	6.4	6.6
DATA FROM CURLL, REFERENCE (9)			
Table I, E_{100} = 14.2 ft-lb	0.394 × 0.197	6.4	6.6
	0.394 × 0.118	4.0	4.6
	0.197 × 0.197	3.2	3.4
	0.118 × 0.118	1.3	1.5
Table II, E_{100} = 72.0 ft-lb	0.394 × 0.197	26.0	23.0
	0.394 × 0.118	12.7	10.6 to 13.7
	0.197 × 0.197	12.6	10.7 to 12.9
	0.118 × 0.118	4.2	4.4
Table III, E_{100} = 44.0 ft-lb	0.394 × 0.197	15.5	15.4
	0.394 × 0.118	8.5	8.0
	0.197 × 0.197	8.3	7.9
	0.118 × 0.118	2.8	2.8

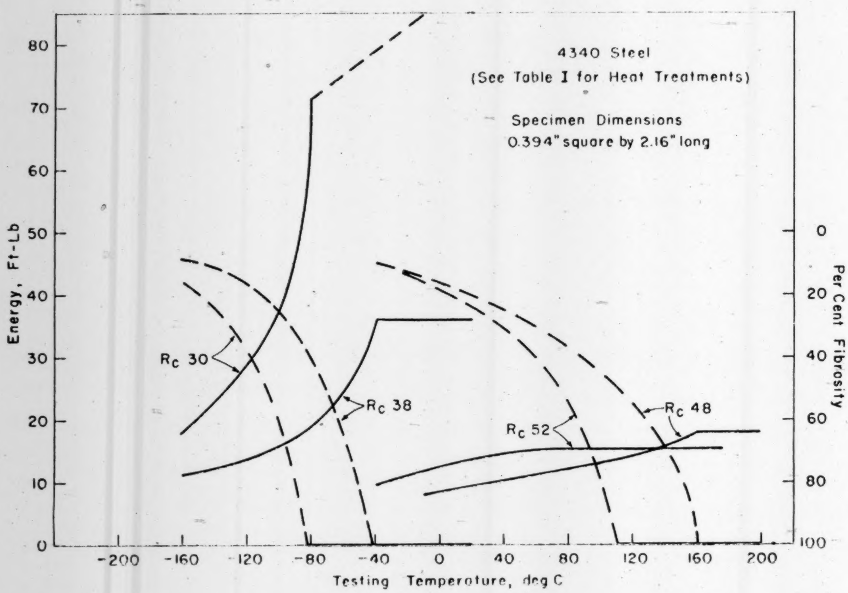


FIG. 5.—Transition Curves for 4340 Steel at Four Strength Levels; Standard Size Square Specimen.

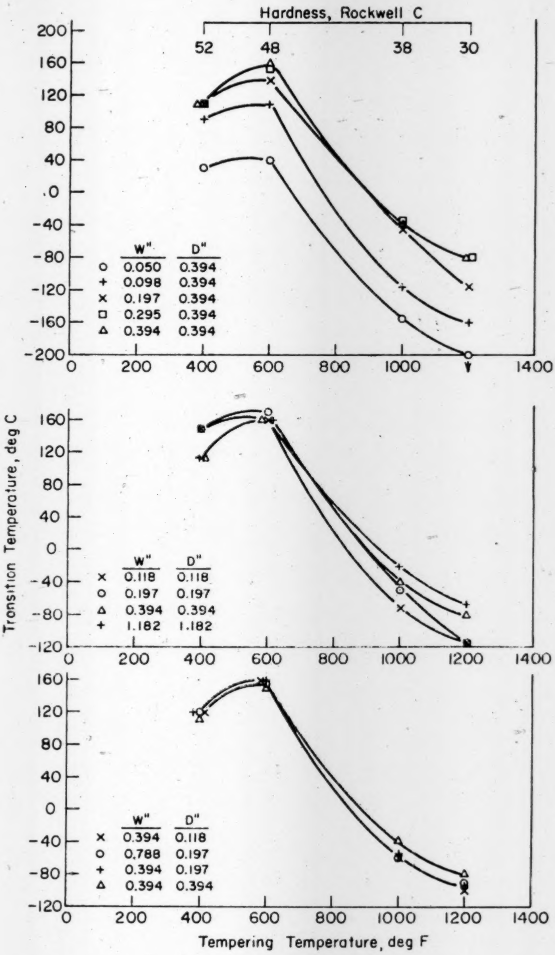


FIG. 6.—Effect of Tempering Temperature and Specimen Geometry on the Transition Temperature of 4340 Steel.

which follows from Eq 9 when $d/D = 1$. When the gross depth was 0.118 or 0.197 in., it was found that the energy could be given by:

$$e_{100} = E_0 \frac{w}{W} \frac{d}{D} + 1.35 \cdot (E_{100} - E_0) \frac{w}{W} \left(\frac{d}{D}\right)^2 \quad \ldots\ldots (11)$$

Table 2 shows a comparison of calculated and actual energy values for various subsize specimens, using either Eq 10 or Eq 11, depending on the depth. As can be seen, agreement is generally satisfactory.

It is recognized that Eq 11 is not in agreement with Eq 10 or Eq 9 when the depth d equals D. It is valid only for one-third or one-half depth bars. The experimental data could also be represented by equations in which the length of the deformed zone was a more complex function of the depth. These in-

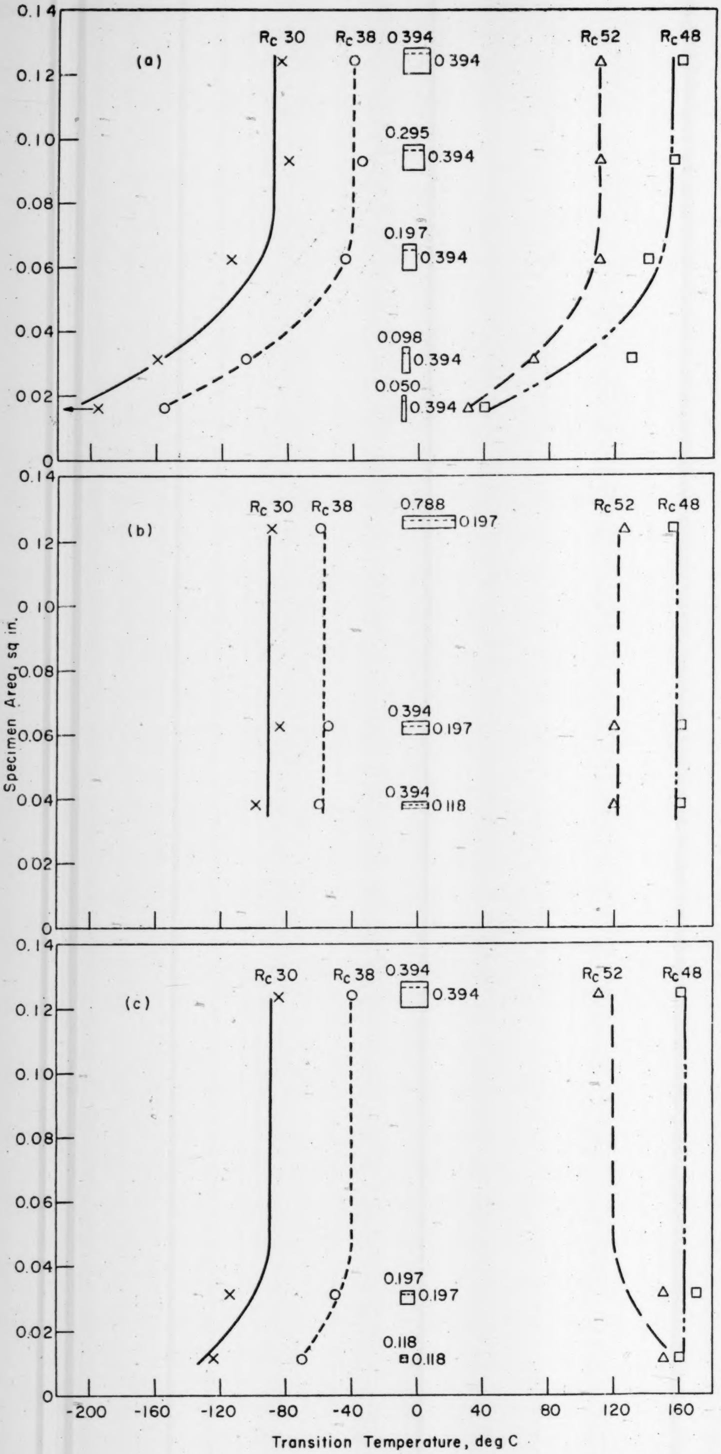


FIG. 7.—Effect of Specimen Size on Transition Temperatures for 4340 Steel Specimens of Differing Geometries.

volved two constants, one or both of which appeared to be a function of the full-size energy level. Because the complexity of the equations reduced their usefulness, it appears best at this time to use Eq 11.

Table 2 shows, in addition to all the data compiled in this investigation, data from Curll's two previous investigations (1,9). All calculations were based on the 0.394-in. square specimen values, and good agreement is indicated. It must be remembered that all comparisons have been made at the 100 per cent fibrosity level, and yet it appears that correlations would be possible at any energy level if the transitional behavior for each strength level and specimen geometry were similar. This, however, is not so, as previous investigations have shown that transition temperatures decrease with specimen size, and it is essential that the effect of both the transitional behavior and the transitional temperature be understood.

Figure 5 illustrates typical transition curves for given specimen sizes at four strength levels. The lowest temperature at which 100 per cent fibrous fracture is found is well defined in each case and is used as the criterion for transition temperatures. It is seen that, in general, the transition temperature increases with hardness, although the highest hardness material did not produce the highest transition temperature. This is more dramatically illustrated in Fig. 6 where, for each specimen geometry, the transition temperature is plotted as a function of tempering temperature for each specimen size.

The effect of specimen area on transition temperature depends on which dimension is reduced, as can be seen in Fig. 6 and more clearly in Fig. 7. When the depth is maintained constant and the width reduced (Fig. 7(*a*)), the transition temperature decreases below widths of 0.197 in. The shapes of the curves for these edge-notch specimens are similar in shape for the different hardness levels. When the depth is reduced, maintaining the width constant at 0.394 in. or increasing it to 0.788 in. (Fig. 7(*b*)), little change in transition temperature is observed. If the dimensions are reduced, keeping the specimen square (Fig. 7(*c*)), the transition temperature decreases with size at low hardnesses and may even increase slightly with decreasing size at the higher hardness level.

Until the magnitude of the effect of specimen size and geometry is fully predictable, correlation of edge-notch subsize specimens should be used with extreme caution. This is necessary, as the fracture of the specimens being compared might be significantly different and erroneously high values would be calculated from the subsize specimens. It could be possible, for example, to test an edge-notch specimen at −160 C and observe 100 per cent fibrous fracture appearance and yet the standard specimen tested at the same temperature would produce a fracture of but 15 per cent fibrosity. If a correlation were required between a subsize edge-notch specimen and a standard bar as, for example, for inspection testing, it would be necessary to test the subsize specimen at some lower temperature (as determined by Fig. 7(*a*)) to ensure that the subsize and standard bars have the same amount of fibrosity in the fracture. Equation 10 could then be used to relate the energies.

In an attempt to determine the cause of the dramatic change in transition temperature for the subsize edge-notch specimens, the fractured specimens were examined. When a full-size specimen is fully ductile, the fracture surface consists of flat fracture in the center and shear lips on each side adjacent to the notch. It has been suggested that the shear lip is essentially constant as the width varies as long as some ductility persists (10). As the specimen width decreases gradually, the amount of flat fracture changes until at the same width the fracture consists only of shear lips and no flat fracture. Since the crystalline fracture is first observed in the flat fracture region, little change in transition temperature would be observed until some critical width is reached; below this width the transition temperature would decrease with further decreases in the width. On the other hand, this explanation may not be valid if the specimen width is at least equal to half of the specimen depth. Here the distance from the surface opposite the notch affects the size of the flat fracture area and hence the transition temperature. Clearly further work is called for.

SUMMARY

Using 4340 steel of four different strength levels, it has been found possible to interrelate Charpy impact values for specimens of differing sizes and geometries.

In agreement with previous work, a linear relationship is obtained between energy to fracture and per cent fibrous fractures. The extrapolated intercept at zero per cent fibrosity varies linearly with specimen area, but is independent of strength level. The energy at 100 per cent fibrosity, which includes the energy to deform the specimen plastically, depends on both the area and strength level. A simple linear relationship exists between this energy and width for standard 0.394-in. depth specimens. When the depth is reduced to one half or one third along with the width, the more complex empirical Eq 11 best describes the energy interrelations.

In addition to the effects of specimen geometry on the energy, there is also an effect on transitional behavior. Although the transition temperature is little affected by reducing the area to half of standard, further reductions of size generally reduce this temperature. The reduction is greatest for reduced-width specimens. Strength level is also a factor, and outsize square specimens of the highest strength level show a slightly higher transition temperature.

This investigation has shown that from the standpoint of energy correlation it is better to maintain a constant depth and reduce the width, but any advantage of ease of correlation is offset by the strong effect of size or transition temperature. Reducing the depth while maintaining the width constant leads to little change in the transition temperature, but here the energy correlations require the use of an empirical equation. It appears that energy correlations are possible, although clearly much work remains to be done.

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A RESISTANCE HEATING FACILITY FOR DETERMINATION OF TENSILE PROPERTIES OF ALLOYS FOR AIRCRAFT AND MISSILES

By W. Kyros,[1] T. W. Gibbs,[1] and C. L. Theberge[1]

Synopsis

Tensile properties were determined for short-time missile applications for a selected group of alloys over temperature ranges extending above the usual limit of application. All tests were performed under carefully controlled heating rates using resistance heating techniques and constant strain rates to provide data for use in the design of missile structures. A resistance heating facility and an extensometer were especially designed for this purpose. The results, obtained on specimens from single 0.125-in. thick sheets parallel to the rolling direction, include the properties of the following alloys over the following temperature ranges:

Alloy	Temperature Range, deg F
2024-T3 aluminum	75 to 800
6061-T6 aluminum	75 to 900
X2020-T6 aluminum	75 to 900
7075-T6 aluminum	75 to 820
AZ31B-H24 magnesium	75 to 700
HK31A-H24 magnesium	75 to 700
B120-VCA titanium	75 to 1500
AISI 316 stainless steel	75 to 1800

An investigation of methods of measuring specimen temperature was undertaken as part of this program in anticipation of future testing at temperature exceeding the 1800 F used for the AISI Type 316 stainless steel. These results indicate that present methods may be extended to 2300 F. They also better defined problem areas for the further extension of the equipment to include the testing of refractory alloys to even higher temperatures.

The design of high-performance aircraft, missiles, and space vehicles has led to an increased need for the determination of stress properties of structural metals under conditions of rapid heating to very high temperatures. Because of high reliability requirements and early delivery dates, many of the newer alloys have not been used in the construction of such specialized vehicles. It was recognized, therefore, that existing information on tensile properties of the more conventional alloys should be supplemented by the evaluation of these properties under conditions similar to those encountered in actual flight.

For the past few years much work has been conducted by several laboratories in the development of test equipment and methods for the purpose of obtaining engineering data under rapid-heating-rate conditions. The following institutions have published extensively in this field: Battelle Memorial Inst. (**1–8**);[2] NASA, Langley, Va. (**9–16**); Naval Ordnance Test Station, Inyokern, China Lake, Calif. (**17–33**); and Southern Research Inst., Birmingham, Ala. (**34–44**). The bibliography also includes much other excellent work (**45–56**). The outstanding problem is the lack of correlation among the laboratories. The ASTM has made some effort to establish tentative standards[3,4] for future work; however, the plight of the design engineer continues to be one of artfully studying and judging data obtained from specialized tests for his own application.

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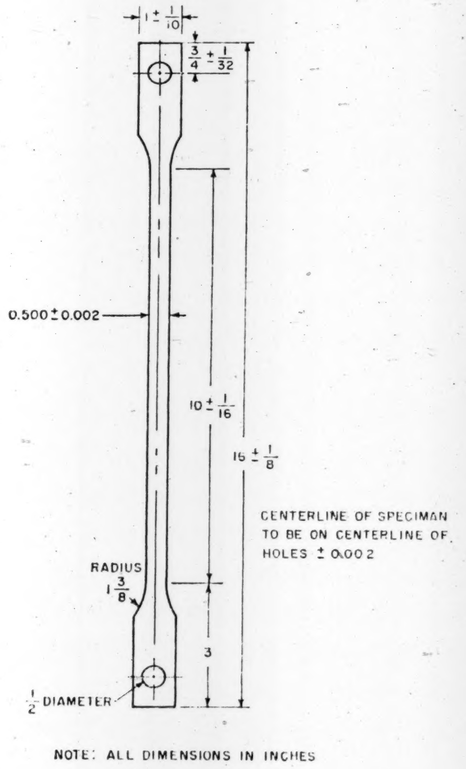
1 ± 1/10
3/4 ± 1/32
0.500 ± 0.002
10 ± 1/16
15 ± 1/8
CENTERLINE OF SPECIMAN TO BE ON CENTERLINE OF HOLES ± 0.002
RADIUS 1 3/8
3
1/2 DIAMETER
NOTE: ALL DIMENSIONS IN INCHES

FIG. 1.—Rapid-Heating-Rate Tension Specimen.

In the development of test equipment, emphasis has been placed on flexibility to accommodate future needs in a rapidly expanding field. The design is adaptable to any standard testing machine to determine accurately basic elastic and plastic properties of metals to 2300 F and may be extended for use at virtually any temperature, provided reliable devices can be developed for accurate measurement of strain, temperature, and temperature control. Inert atmosphere and vacuum chambers may be conveniently incorporated into the system. Suitable modifications to plastic and ceramic testing facilities, to allow testing under conditions more similar to reentry than present techniques allow, also seem feasible.

It is noted that the selected test conditions allowed the comparison of conventional short hold time with very short hold time at temperature; but more important, all tests were performed at a constant, controlled strain rate. Data on constant strain rate are not plentiful, and it is evident that such data are required for efficient design in the plastic region of deformation.

MATERIALS, EQUIPMENT, AND TEST PROCEDURE

Materials and Specimen Design:

All materials used in this evaluation program were purchased from commercial suppliers as rolled sheet stock

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

[3] Recommended Practice for Conducting Creep and Creep-Rupture Tension Tests of Metallic Materials Under Conditions of Rapid Heating and Short Times (E 150), *1961 Book of ASTM Standards*, Part 3.

[4] Recommended Practice for Tension Tests of Metallic Materials at Elevated Temperatures with Rapid Heating and Conventional or Rapid Strain Rates (E 151), *1961 Book of ASTM Standards*, Part 3.

TABLE 1.—MATERIALS TESTED—CHEMICAL COMPOSITION AND HEAT TREATMENT[a].

Aluminum Alloys:

Alloy and Temper	Element, per cent												Temperature Range of Tests
	Silicon	Iron	Copper	Maganese	Magnesium	Chromium	Zinc	Titanium	Lithium	Cobalt	Other	Aluminum	
2024-T3	0.05	0.34	4.25	0.61	1.42	<0.05	<0.05	...	...	...	0.016	balance	room temperature to 800 F
Nominal composition	...	...	4.5	0.6	1.5	...	...	...	...	...	...	balance	
6061-T6 (bare)	0.58	0.37	0.29	0.10	0.84	0.26	<0.10	0.024	...	...	...	balance	room temperature to 900 F
Nominal composition	0.6	...	0.25	...	1.0	0.25	...	...	...	...	...	balance	
X2020-T6	0.12	0.27	3.9	0.51	...	...	...	0.037	1.1	0.10	0.022	balance	room temperature to 900 F
Nominal composition	0.4 max	0.4 max	4.5	0.55	0.03 max	...	0.25 max	0.1 max	1.3	0.22	0.15 max	balance	
7075-T6	0.10	0.165	1.30	0.115	2.10	0.22	5.20	0.078	...	...	...	balance	room temperature to 820 F
Nominal composition	0.5 max	0.7 max	1.6	0.3 max	2.5	0.29	5.6	0.2 max	...	...	0.15 max	balance	

Magnesium Alloys:

Alloy and Temper	Element, per cent												Temperature Range of Tests
	Silicon	Iron	Copper	Manganese	Calcium	Thorium	Zinc	Zirconium	Aluminum	Nickel	Other	Magnesium	
AZ31B-H24	0.3	<0.005	0.003	0.35	<0.04	...	...	1.00	3.05	<0.005	<0.3	balance	room temperature to 700 F
Nominal composition	0.1 max	0.005 max	0.05 max	0.2 min	0.04 max	...	1.0	...	3.0	0.005 max	0.30 max	balance	
HK31A-H24	...	...	...	...	...	3.08	...	0.58	...	...	<0.3	balance	room temperature to 700 F
Nominal composition	...	...	...	...	...	3.25	...	0.70	...	..	0.3 max	balance	

Titanium Alloys:

Alloy and Temper	Element, per cent							Temperature Range of Tests
	Carbon	Nitrogen	Hydrogen	Vanadium	Chromium	Aluminum	Titanium	
B120-VCA	0.03	0.019	0.021	12.9	11.8	3.8	balance	room temperature to 1500 F
Nominal composition	...	...	...	13.0	11.0	3.0	balance	

Other Alloys:

Alloy and Temper	Element, per cent								Temperature Range of Tests
	Carbon	Manganese	Sulfur	Silicon	Chromium	Nickel	Molybdenum	Iron	
Type 316 stainless steel	0.03	1.70	0.013	0.20	17.17	12.96	2.15	balance	room temperature to 1800 F
Nominal composition	0.08 max	2.0 max	0.03 max	0.75 max	17.0	12.5	2.5	balance	

[a] 0.125-in. sheet material specimens parallel to rolling direction.

0.125 in. thick. A sufficient quantity of each material was obtained so that all test specimens were taken from the same sheet parallel to the rolling direction (Fig. 1). Chemical compositions of each alloy tested are given in Table 1.

Heating Equipment:

In the performance of the tests, the rapid-heating rates and temperature control were achieved with equipment designed to use resistance heating and usable with any electrically conductive material (Fig. 2). It consists of a power supply, an Ignitron control circuit, a programming function generator, and a step-down transformer, with the necessary measuring instruments. The power is supplied through a 440-v, 800-amp bus duct to the Ignitron control cubicle. Any desired heating rate or temperature-time program can be drawn on the function generator (described below) from which the signal may be compared indirectly to that from the actual specimen temperature obtained with a thermocouple by a retransmitting slide wire in the temperature recorder. These two signals are compared at a summing junction and the error signal controls the power at the Ignitron tubes. The output from these tubes, which may vary from 0 to 440 v, is stepped down to much lower voltages and higher currents in a 64-kva transformer, and then passed through the specimen. The total capacity of the system based on the Ignitron limit is 352 kva.

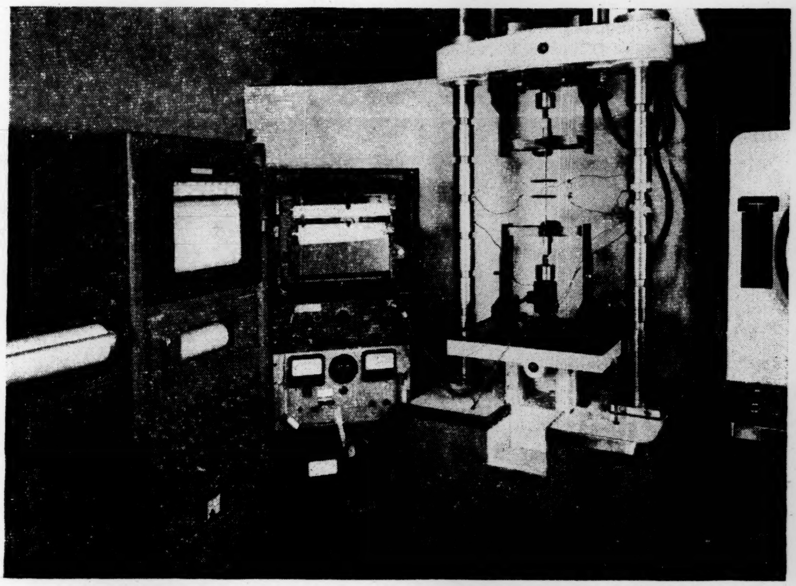

FIG. 2.—Equipment Used for Rapid-Heating Rates and Temperature Control.

The function generator is essentially a slide wire potentiometer with a silvered-ink graph. The desired temperature-time curve is drawn with silvered ink on graph paper and is fastened to a cylindrical drum. This curve acts as the movable arm of the potentiometer.

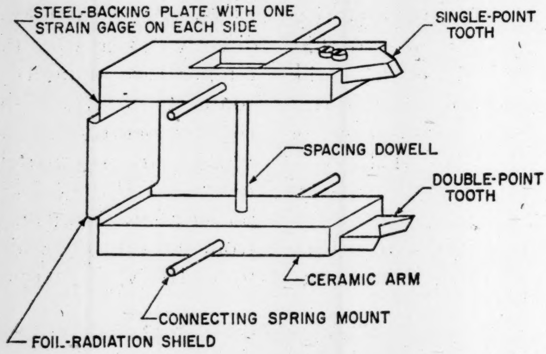
STEEL-BACKING PLATE WITH ONE STRAIN GAGE ON EACH SIDE
SINGLE-POINT TOOTH
SPACING DOWELL
DOUBLE-POINT TOOTH
CERAMIC ARM
CONNECTING SPRING MOUNT
FOIL-RADIATION SHIELD

FIG. 3.—Half-Unit of Rapid-Heating-Rate Extensometer.

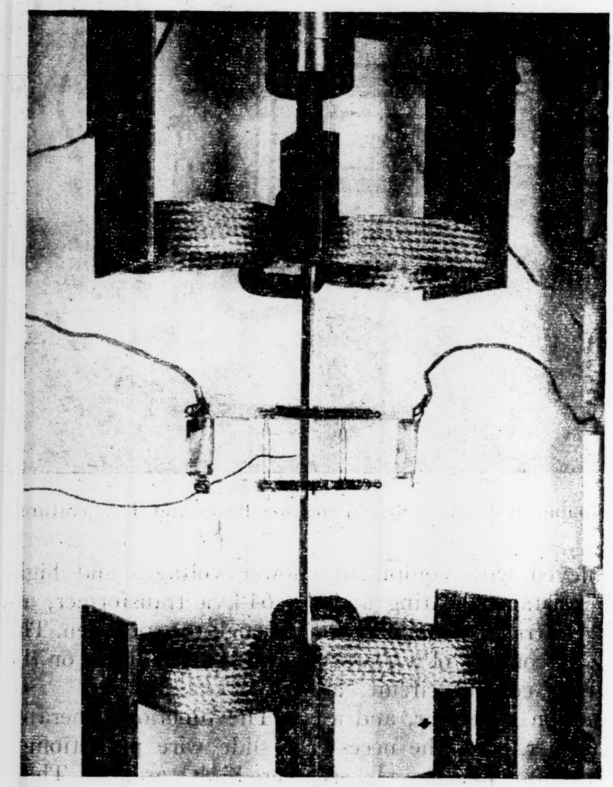

FIG. 4.—Units Held Together and in Contact with Specimen.

Extensometer:

A specially designed nonconducting extensometer capable of measuring strain to an accuracy of 0.000020 in. per in. was used for this work. This instrument consists of two identical halves, one of which is shown in Fig 3. Four springs hold the two units together and in contact with the specimen (Fig. 4). Temperature-compensating strain gage bridges are mounted on steel strips at the end of insulating ceramic areas sufficiently far from the specimen and shielded from radiation to maintain the temperature at a low enough level (less than 150 F) that temperature effect on strain gages is negligible. The signal from this extensometer feeds into a high-gain amplifier which in turn feeds the amplified signal into a two-pen recording system which simultaneously records load-time and strain-time curves.

Tensile Stress-Strain Data:

The specimens were heated under no load at a constant temperature rate of 50 F per sec (±3 F to the test temperature); held for the reported times; and strained at a constant-controlled rate through the yield stress until the maximum stress had been achieved. Tests were conducted at the reported test temperatures after the prescribed hold times and the strain rate controlled at a nominal 0.004 in. per in. per min. Strain rate control was exercised throughout the duration of the test by manually maintaining the strain recording pen on a predrawn strain-time slope on the chart paper. The load was applied by a Baldwin FGT mechanical testing machine which used an SR-4 cell for the measurement of loads, and stress-strain curves were obtained by cross-plotting the simultaneous load-time and strain-time data.

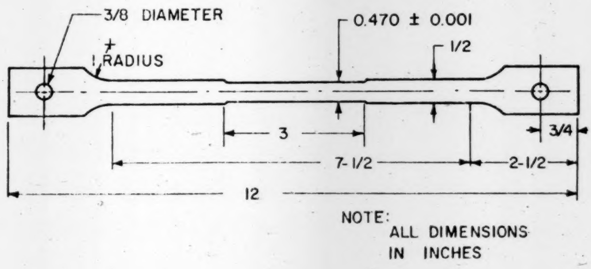
3/8 DIAMETER
1 RADIUS
0.470 ± 0.001
1/2
3
3/4
7-1/2
2-1/2
12
NOTE:
ALL DIMENSIONS
IN INCHES

FIG. 5.—Rapid-Heating-Rate Tension Specimen B.

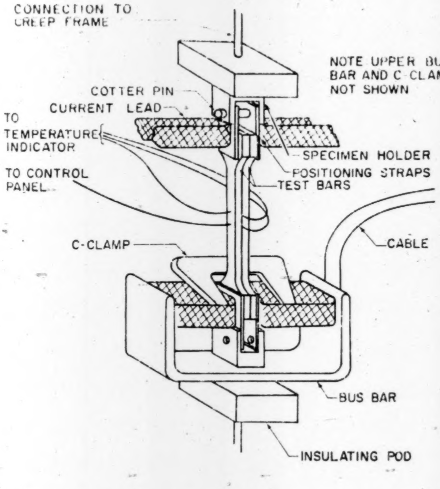
CONNECTION TO CREEP FRAME
NOTE UPPER BUS BAR AND C CLAMP NOT SHOWN
COTTER PIN
CURRENT LEAD
TO TEMPERATURE INDICATOR
SPECIMEN HOLDER
POSITIONING STRAPS
TEST BARS
TO CONTROL PANEL
CABLE
C-CLAMP
BUS BAR
INSULATING POD

FIG. 6.—"Sandwich" Specimen in Position for Determination of Transverse Thermal Gradients.

Evaluation of Rapid-Heating-Rate Temperature Measurement Methods and Specimen Temperature Gradients:

Prior to the test program on each of the eight materials tested, an indication of the temperature gradient was obtained across the gage length of the specimen at the maximum anticipated test temperature.

An additional investigation was undertaken to evaluate chromel-alumel thermocouple performance over the temperature range from 1000 F to 2300 F. Haynes Alloy No. 25, also known as L605, a ductile cobalt-base alloy was used throughout this phase.

Two shorter specimens not previously

used were investigated for gradients as follows: a 12-in. long specimen known as rapid-heating-rate specimen B (Fig. 5) having a milled central section to ensure fracture within the gage length, and an 8-in. long specimen of similar gage section. Both were prepared and tested with and without the milled central section to determine its influence on the longitudinal thermal gradient

TABLE 2.—RAPID-HEATING-RATE TENSILE PROPERTIES OF 2024-T3 ALUMINUM AT ELEVATED TEMPERATURES AND A STRAIN RATE OF 0.004 IN. PER IN. PER MIN FOR VARIOUS HOLD TIMES.

Test Temperature, deg F	Hold Time, sec	Yield Strength (0.2 per cent Offset), psi	Tensile Strength, psi	Modulus of Elasticity, *E*, psi
Room temperature (75)	...	53 100	66 800	10.4 × 10^6
Room temperature	...	53 200	69 700	10.2
400	1 800	40 500	47 100	8.9
	1 800	42 900	49 000	8.8
	1 800	45 700	50 200	8.7
	120	39 400	46 500	7.7
	120	38 000	45 000	7.3
	60	40 100	48 000	7.8
	30	39 500	47 000	7.8
	30	40 300	47 900	7.6
	15	39 100	48 200	8.0
600	1 800	12 000	12 300	8.0
	120	18 800	19 300	5.2
	120	18 100	18 600	5.6
	65	19 000	19 100	6.5
	60	18 400	18 600	5.9
	65	19 000	19 000	5.9
	30	18 600	19 800	6.0
	30	18 400	20 000	6.3
	30	20 400	20 500	6.0
700	1 800	7 560	7 700	6.1
	1 800	8 300	8 410	5.6
	120	11 700	12 000	5.3
	120	11 600	11 600	5.9
	120	11 800	12 400	5.8
	60	12 400	12 500	4.9
	65	12 800	13 100	5.9
	40	12 400	12 900	4.8
	30	11 900	12 100	5.1
	30	12 500	12 700	4.3
	15	11 500	11 700	5.8
800	1 800	3 380	3 420	2.7
	1 800	3 160	3 400	3.7
	1 800	3 170	3 520	3.7
	1 800	3 250	3 450	3.4
	1 800	2 970	3 060	3.4
	40	4 250	4 280	3.1
	40	4 350	4 820	3.8

Chromel-alumel thermocouples of No. 28 gage wire were used throughout the investigation. Heavier gage wires, although stronger and less likely to break, were not used because of the cooling effect of conduction along the wire. Temperatures were measured at $\frac{1}{4}$-in. intervals on alternating sides of the bar by thermocouples, each wire of which was separately spot welded to the specimen surface $\frac{1}{8}$-in. apart parallel to the specimen axis. This method is used frequently (**17,56**), for it decreases the response time of the thermocouple to temperature change, an important consideration in rapid-heating-rate testing.

TABLE 3.—RAPID-HEATING-RATE TENSILE PROPERTIES OF 6061-T6 ALUMINUM AT ELEVATED TEMPERATURES AND A STRAIN RATE OF 0.004 IN. PER IN. PER MIN FOR 30- AND 1800-SEC HOLD TIMES.

Test Temperature, deg F	Hold Time, sec	Yield Strength (0.2 Per Cent Offset), psi	Tensile Strength, psi	Modulus of Elasticity, E, psi
Room temperature.......	...	42 700	47 400	10.1×10^6
	...	42 200	47 200	10.1
300....................	1 800	36 000	36 100	9.8
	1 800	35 700	36 000	9.4
	30	36 400	36 700	9.7
	30	35 700	36 300	9.7
500....................	1 800	18 600	18 800	8.2
	1 800	18 400	18 600	7.8
	30	21 000	21 600	8.5
	30	20 300	20 800	8.1
600....................	1 800	8 100	8 100	7.0
	1 800	8 300	8 500	7.1
	30	12 200	12 500	7.1
	30	12 300	12 400	6.9
700....................	1 800	4 300	4 300	5.7
	1 800	3 900	4 100	5.2
	30	6 100	6 100	6.0
	30	5 900	5 900	5.6
800....................	1 800	2 200	2 500	5.0
	1 800	2 100	2 200	3.6
	30	2 900	3 000	4.9
	30	2 700	2 800	3.9
900....................	1 800	1 530	1 540	2.9
	1 800	1 520	1 620	2.6
	30	1 430	1 640	2.0
	30	1 480	1 510	2.5

Tests were conducted to determine temperature measurement reproducibility and longitudinal thermal gradients at five temperatures: 1000, 1400, 1600, 2000, and 2300 F. Testing was limited to four test conditions: (1) type B specimen (milled) without extensometer, (2) type B specimens (milled) with extensometer, (3) type C specimen (not milled) without extensometer, and (4) type C specimen (milled) without extensometer. Instrumented bars were tested in a modified creep frame using the fixtures shown in Fig. 6.

In an attempt to measure transverse thermal gradients, a method used in earlier work at Battelle was tried (**3**). Two 12-in. specimens were bolted together to form a single specimen, shown in place in the creep frame in Fig. 6. Two pairs of thermocouples were welded to one specimen, one thermocouple on each side at center and one 1 in. away. When bolted to the other specimen, this produced, in effect, a single specimen with thermocouples imbedded in the center and mounted on the outside. The difference between inner and outer

TABLE 4.—RAPID-HEATING-RATE TENSILE PROPERTIES OF X2020-T6 ALUMINUM AT ELEVATED TEMPERATURES AND A STRAIN RATE OF 0.004 IN. PER IN. PER MIN FOR A 30-SEC HOLD TIME.

Test Temperature, deg F	Hold Time, sec	Yield Strength (0.2 Per Cent Offset), psi	Tensile Strength, psi	Modulus of Elasticity, E, psi
Room temperature	...	80 700	82 700	11.1×10^6
	...	79 700	82 400	10.9
600	30	20 500	20 600	8.4
	30	19 500	19 700	8.0
800	30	5 000	5 100	4.8
	30	4 500	4 500	4.8
900	30	1 900	1 900	3.8
	30	1 300	1 500	2.9

TABLE 5.—RAPID-HEATING-RATE TENSILE PROPERTIES OF 7075-T6 ALUMINUM AT ELEVATED TEMPERATURES AND A STRAIN RATE OF 0.004 IN. PER IN. PER MIN FOR VARIOUS HOLD TIMES.

Test Temperature, deg F	Hold Time, sec	Yield Strength (0.2 Per Cent Offset), psi	Tensile Strength, psi	Modulus of Elasticity, E, psi
Room temperature	...	74 400	74 600	10.3×10^6
300	1 800	57 000	57 000	8.9
	1 800	56 300	56 300	8.9
	60	58 000	58 200	8.8
	30	58 300	59 900	9.2
500	1 800	17 900	17 900	8.6
	1 800	17 900	18 100	8.3
	30	24 700	25 500	7.4
	30	25 500	25 900	7.4
700	1 800	5 140	5 420	6.3
	1 800	5 740	5 750	5.8
	1 800	5 000	5 150	6.2
	1 800	5 300	5 400	5.2
	30	5 650	5 990	6.0
685	30	6 100	6 340	5.1
800	30	4 160	4 260	4.7
820	30	3 580	3 700	3.8
	30	3 620	3 680	4.4

thermocouple readings gives the limits of the transverse gradient at that point. Thermocouples were also wired onto various parts of the extensometer (Fig. 3).

TABLE 6.—RAPID-HEATING-RATE TENSILE PROPERTIES OF AZ31B-H24 MAGNESIUM AT ELEVATED TEMPERATURES AND A STRAIN RATE OF 0.004 IN. PER IN. PER MIN FOR VARIOUS HOLD TIMES.

Test Temperature, deg F	Hold Time, sec	Yield Strength (0.2 Per Cent Offset), psi	Tensile Strength, psi	Modulus of Elasticity, E, psi
Room temperature	...	30 900	40 100	6.4×10^6
300	1 800	11 500	14 500	3.7
	1 800	12 400	14 900	3.5
	120	9 700	14 700	3.1
	120	11 400	13 900	3.8
	120	11 500	13 400	3.8
	60	10 400	14 800	3.2
	60	11 900	14 700	3.5
	30	12 600	17 000	3.3
	30	10 300	15 100	3.3
	30	12 700	15 700	3.7
500	1 800	4 140	4 490	...
	30	3 370	4 130	1.6
	30	5 050	5 110	1.5
	30	3 370	4 500	1.6
600	1 800	2 000	2 480	1.3
	1 800	2 090	2 200	1.2
	1 800	2 220	2 370	1.3
	1 800	1 650	2 480	1.1
	1 800	1 400	2 400	1.2
	120	1 270	1 990	1.3
	120	1 240	1 960	0.9
	60	1 210	1 860	1.0
	60	1 140	2 010	1.1
	30	1 760	2 350	1.0
	30	1 250	2 150	1.2
	30	1 250	2 230	1.0
700	1 800	840	950	0.6
	1 800	530	1 020	0.9
	1 800	750	900	0.7
	40	540	990	0.9
	40	950	1 300	0.8
	15	520	990	0.9

DISCUSSION

Results of Stress-Strain Tests:

Many data have been published on tensile properties of alloys after hold times at temperature for times of $\frac{1}{2}$ hr and upward. Evidence of increased strength with decreased hold times is borne out in several instances as is shown by the results of almost 200 tests on eight alloys which are presented in Tables 2 to 9. These data have not only extended the useful range of each alloy, but have permitted weight savings in the design of structures. Typical stress-strain curves, presented in Figs. 7 to 11, indicate the nature of the temperature and elastic and plastic responses of the alloys under constant strain rate conditions and allow any appropriate yield criteria to be selected.

Figures 7 and 8 contain the stress-strain curves of the 2024-T3, 6061-T6, X2020-T6, and 7075-T6 aluminum alloys; at 400 F, the 2024-T3 alloy responded with higher yield stresses for the 1800-sec hold time than for the <120-sec hold condition. This can perhaps be explained on the basis of increased age hardening at this temperature because the original 2024 material was not subjected to an artificial aging cycle but was naturally aged. At the higher temperatures, overaging occurred rapidly and higher strengths resulted for the shorter hold times. The increased age hardening phenomena were not exhibited in the other aluminum alloys in that all are in an artificially aged state and, in general, shorter hold times result in higher strengths.

TABLE 7.—RAPID-HEATING-RATE TENSILE PROPERTIES OF HK31A-H24 MAGNESIUM AT ELEVATED TEMPERATURES AND A STRAIN RATE OF 0.004 IN. PER IN. PER MIN FOR VARIOUS HOLD TIMES.

Test Temperature, deg F	Hold Time, sec	Yield Strength (0.2 Per Cent Offset), psi	Tensile Strength, psi	Modulus of Elasticity, E, psi
Room temperature	...	25 800	31 300	6.3×10^6
	...	25 500	31 200	6.4
300	1 800	20 700	22 400	5.0
	120	20 600	22 100	5.3
	120	20 500	22 000	5.6
	120	20 700	21 600	5.6
	30	20 500	21 700	5.5
600	1 800	4 000	4 520	3.0
	1 800	4 500	4 940	2.9
	120	3 040	3 870	2.4
	120	4 360	5 290	2.9
	120	5 190	5 530	2.7
	120	4 610	5 460	3.0
	130	4 400	6 360	3.3
	120	4 120	5 760	2.9
	60	4 760	5 300	2.9
	60	3 840	4 860	3.3
	60	4 460	5 260	3.2
	60	4 440	5 290	2.6
	30	4 830	5 380	3.5
	30	4 420	4 680	3.3
	30	5 140	5 260	2.9
	30	4 500	4 590	2.1
700	100	1 310	1 760	1.0
	100	1 440	1 790	0.9
	100	1 310	1 500	0.8
	60	1 010	1 500	1.0
	30	1 480	2 130	1.0
	30	1 480	1 650	0.8
	15	1 770	1 960	0.9
	15	1 150	1 700	0.9

The variations of yield strength, ultimate strength, and modulus of elasticity are presented in Figs. 12, 13, and 14, respectively, for all the aluminum alloys. It is apparent that almost all identity among the alloys is lost above 800 F where all of the lower temperature strength mechanisms are lost.

The magnesium alloys HK31A-H24 and AZ31B-H24 seem to be relatively

TABLE 8.—RAPID-HEATING-RATE TENSILE PROPERTIES OF B120-VCA TITANIUM AT ELEVATED TEMPERATURES AND A STRAIN RATE OF 0.004 IN. PER IN. PER MIN FOR VARIOUS HOLD TIMES.

Test Temperature, deg F	Hold Time, sec	Yield Strength (0.2 Per Cent Offset), psi	Tensile Strength, psi	Modulus of Elasticity, *E*, psi
Room temperature	. . .	130 600	130 600	14.3×10^6
500	1 800	105 000	110 000	13.0
	1 800	106 000	113 000	12.0
	30	105 000	113 000	12.9
	30	105 000	113 000	11.8
1000	1 800	81 400	83 200	10.3
	1 800	79 800	81 300	10.1
	120	81 900	82 000	11.2
	60	82 200	82 500	10.8
	30	81 100	81 700	10.9
	30	82 900	82 900	10.9
1250	1 800	25 300	27 100	7.8
	1 800	24 000	27 900	7.9
	30	21 300	21 300	8.6
	30	25 500	25 700	8.1
1500	1 800	9 200	10 000	5.8
	1 800	7 600	8 600	4.6
	30	7 100	8 460	5.8
	30	7 350	9 320	4.4
	30	8 900	9 470	4.6
	30	9 700	9 970	4.6

TABLE 9.—RAPID-HEATING-RATE TENSILE PROPERTIES OF AISI TYPE 316 STAINLESS STEEL AT ELEVATED TEMPERATURES AND A STRAIN RATE OF 0.004 IN. PER IN. PER MIN FOR 30- AND 1800-SEC HOLD TIMES.

Test Temperature, deg F	Hold Time, sec	Yield Strength (0.2 Per Cent Offset), psi	Tensile Strength, psi	Modulus of Elasticity, *E*, psi
Room temperature	. . .	30 300	76 800	27.7×10^6
	. . .	29 100	76 500	28.0
500	1 800	22 300	63 500	26.2
	1 800	22 700	63 500	25.7
1000	1 800	18 200	57 400	22.9
	. . .	. . .	. . .	. . .
1400	1 800	11 600	19 500	17.9
	1 800	13 900	20 600	18.3
	30	12 900	23 700	18.2
	30	12 700	21 900	17.3
1600	1 800	10 900	12 600	13.0
	1 800	10 800	12 000	14.0
	1 800	9 600	10 700	11.6
	30	9 650	12 100	13.0
	30	9 750	11 400	13.9
1800	1 800	4 860	6 390	10.5
	1 800	5 300	7 040	9.8
	30	4 900	6 600	9.5
	30	5 330	6 080	9.5

insensitive to the effects of the hold times used in this program. The hint of an effect may be exhibiting itself, however, in Fig. 15, where strength for the short hold times are slightly higher than the longer hold times in the temperature range from 400 to 600 F. This might suggest the possibility that more pronounced effects would manifest themselves for much shorter hold times and higher heating rates. Figure 16 shows the variation of the modulus of elasticity with temperature for the magnesium.

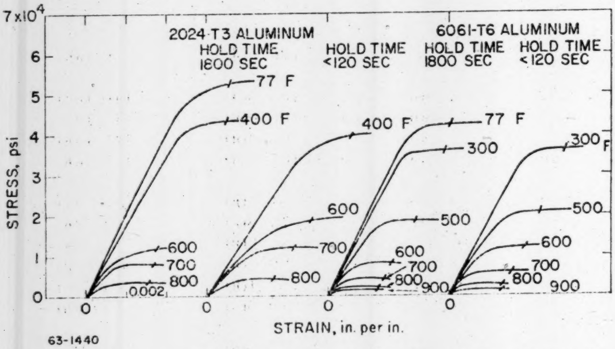

FIG. 7.—Typical Tensile Stress-Strain Curves for 2024-T3 and 6061-T6 Aluminum Alloys at Hold Times of 120 and 1800 sec and Their Variation with Temperature.

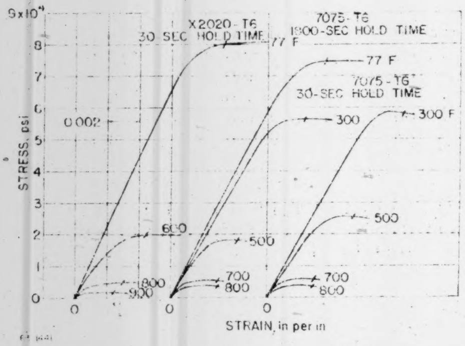

FIG. 8.—Typical Tensile Stress-Strain Curves for X2020-T6 and 7075-T6 Aluminum Alloys at Hold Times of 30 and 1800 sec and Their Variation with Temperature.

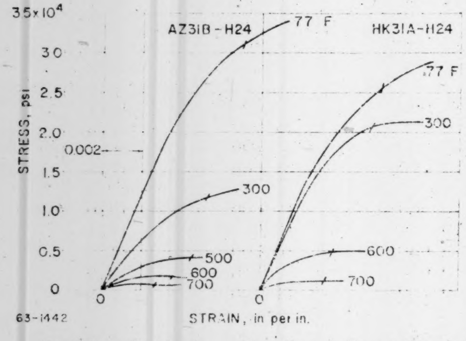

FIG. 9.—Typical Tensile Stress-Strain Curves for AZ31B-H24 and HK31A-H24 Magnesium Alloys and Their Variation with Temperature.

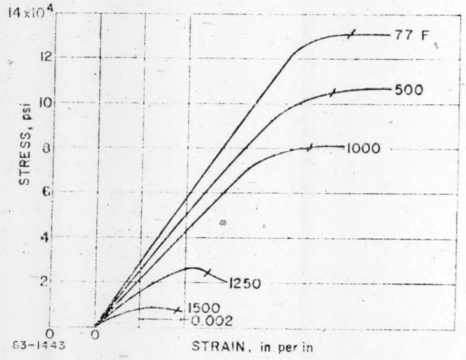

FIG. 10.—Typical Tensile Stress-Strain Curves for B120-VCA Titanium Alloy and Their Variation with Temperature.

The B120-VCA titanium alloy and the AISI Type 316 stainless steel were both in the annealed condition initially, and drastic effects of hold time would not be expected and the results show that this is true. An inference may be made on the basis of these results with respect to particular experimental questions: any longitudinal or transverse temperature gradient across the gage length resulting from a shorter hold time is not significant with respect to the properties obtained.

Figure 17 shows that the stress advantage of AISI Type 316 stainless steel over titanium becomes apparent over 1400 F; however, the weight advantage of titanium below this temperature would be of consideration in a design application. Figure 18 clearly shows the superiority of AISI Type 316 steel over titanium with respect to the modulus of elasticity.

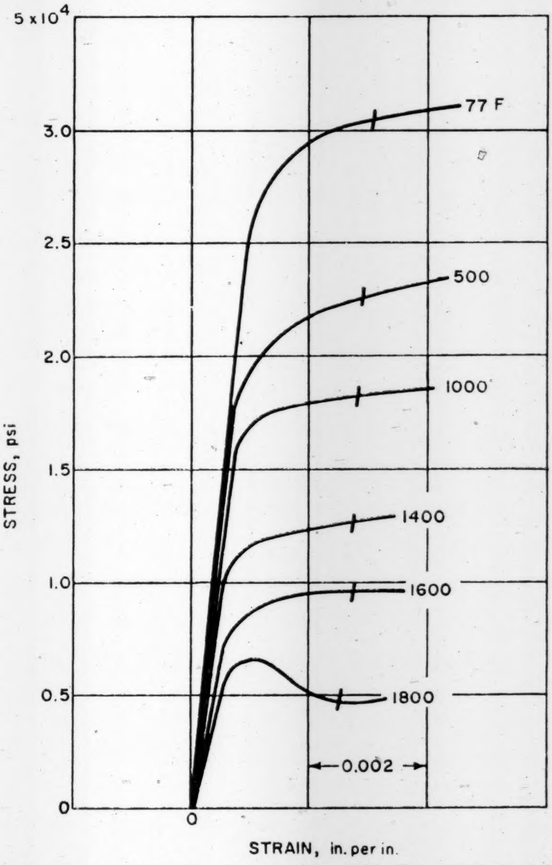

FIG. 11.—Typical Tensile Stress-Strain Curves for AISI Type 316 Stainless Steel and Their Variation with Temperature.

Measurement of Elastic Modulus:

The determination of modulus of elasticity in tension, even to satisfy the practical requirements, requires rather a precise instrumentation system and a highly skilled operator capable of performing these refined measurements on a repetitive basis. The possible sources of errors in the measurement of modulus, for both room- as well as elevated-temperature testing, provide a guide in taking various steps for their elimination: (1) inherent error in strain measuring system, (2) extensometer out of calibration, (3) specimens not machined to close enough tolerances, (4) excessive temperature gradients along gage length, (5) improper mounting of extensometer, (6) inadequate rate control, (7) interpretation of stress-strain curve, and (8) nonaxial loading.

With respect to point 1 above as a source of error, Fig. 19 shows the relationship between the maximum error in the modulus of elasticity versus the maximum available elastic strain as derived from the stress-strain curve. As the test temperature is increased, the available elastic strain (or elastic limit) is correspondingly decreased to the point where the permissible error in the extensometer system approaches the same order of magnitude as the total available elastic strain. The error curve for the class B-1 extensometer in Fig. 19 il-

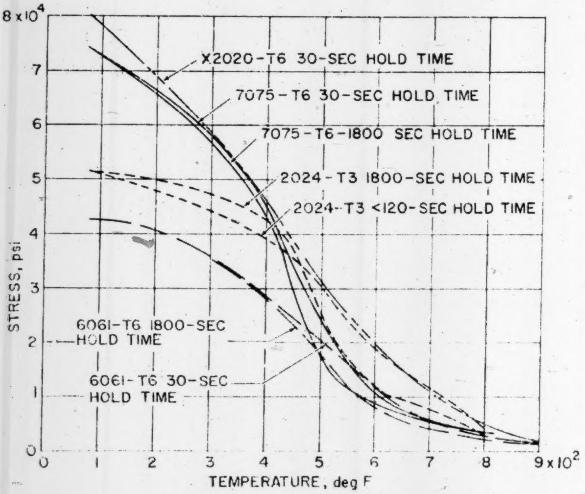

FIG. 12.—Tensile Yield Stress for 2024-T3, 6061-T6, X2020-T6, and 7075-T6 Aluminum Alloys at Elevated Temperatures for Hold Times to 1800 sec and a Strain Rate of 0.004 in. per in. per min.

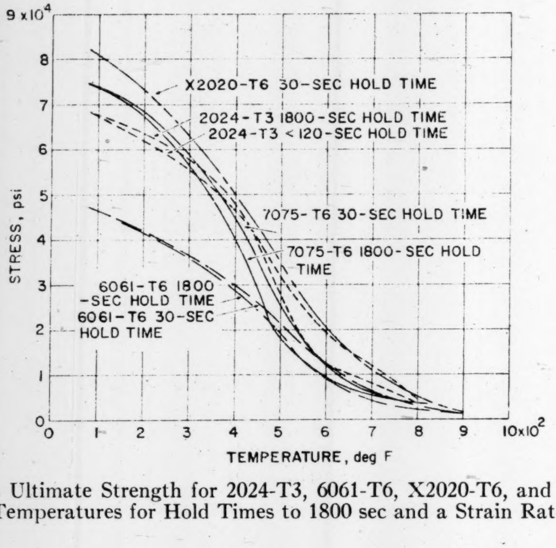

FIG. 13.—Tensile Ultimate Strength for 2024-T3, 6061-T6, X2020-T6, and 7075-T6 Aluminum Alloys at Elevated Temperatures for Hold Times to 1800 sec and a Strain Rate of 0.004 in. per in. per min.

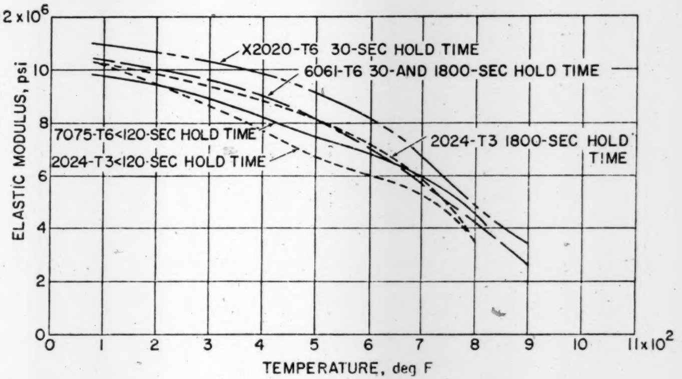

FIG. 14.—Tensile Modulus of Elasticity for 2024-T3, 6061-T6, X2020-T6, and 7075-T6 Aluminum Alloys at Elevated Temperatures for Hold Times to 1800 sec and a Strain Rate of 0.004 in. per in. per min.

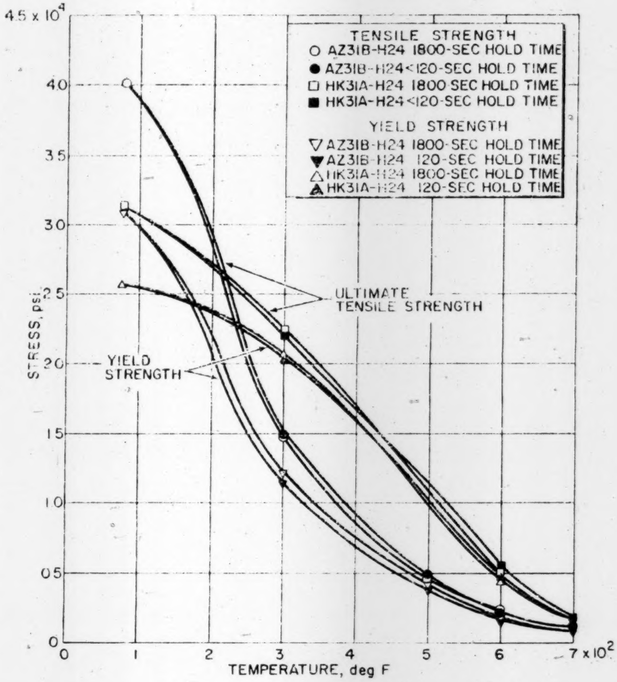

FIG. 15.—Tensile Yield Strength and Ultimate Strength for HK31A-H24 and AZ31B-H24 Magnesium Alloys at Elevated Temperatures for Hold Times to 1800 sec and a Strain Rate of 0.004 in. per in. per min.

lustrates the worst case of modulus error when using an ASTM B-1 classification strain-measuring system.[5] Such a limit is usually acceptable for general tension testing. However, for missile applications such errors are unacceptable; hence the development of a special extensometer

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[5] Method of Verification and Classification of Extensometers (E 83), *1961 Book of ASTM Standards*, Part 3.

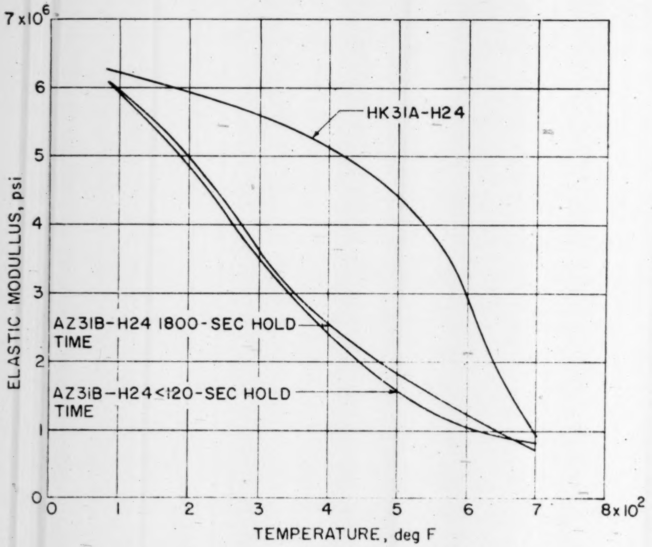


FIG. 16.—Tensile Modulus of Elasticity for HK31A-H24 and AZ31B-H24 Magnesium Alloys at Elevated Temperatures and for Hold Times to 1800 sec and a Strain Rate of 0.004 in. per in. per min.

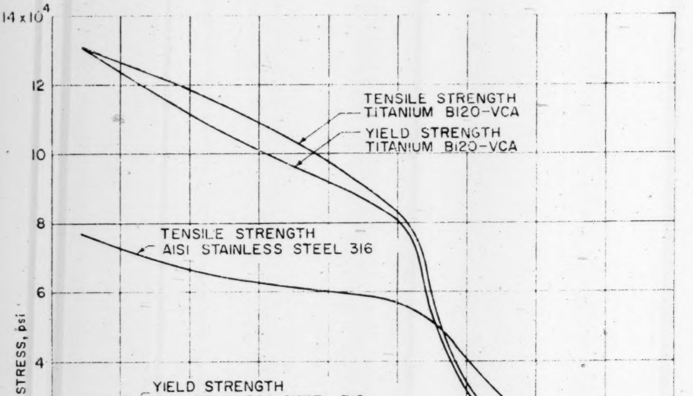


FIG. 17.—Tensile Yield Strength and Ultimate Strength for B120-VCA Titanium Alloy and AISI Type 316 Stainless Steel at Elevated Temperatures for Hold Times to 1800 sec and a Strain Rate of 0.004 in. per in. per min.

system was undertaken and the error curve is included in Fig. 19. As an example for the determination of the maximum error that may be expected from the Avco RAD extensometer system alone, consider AZ31B-H24 alloy at 600 F having available elastic strains (see Fig. 9) ranging from 0.00015 in. per in.

to 0.0006 in. per in. From Fig. 19, it is seen that the maximum modulus error ($\Delta E/E \times 100$) is +12 per cent, −15 per cent for the 0.00015 in. per in. (ϵ_e) level, and ±3 per cent at the 0.0006-in. per in. ϵ_e level.

Two steps were taken to minimize point 2 above regarding calibration as a source of error. First, the extensometer was calibrated at room temperature before and after a series of tests; second, the thermal expansion of each bar was recorded and compared to the known thermal expansion of the particular material in question prior to each test. Although this is an indirect means of calibrating at elevated temperature conditions, reasonable assurance was derived to indicate that the "hot" extensometer was not significantly out of calibration.

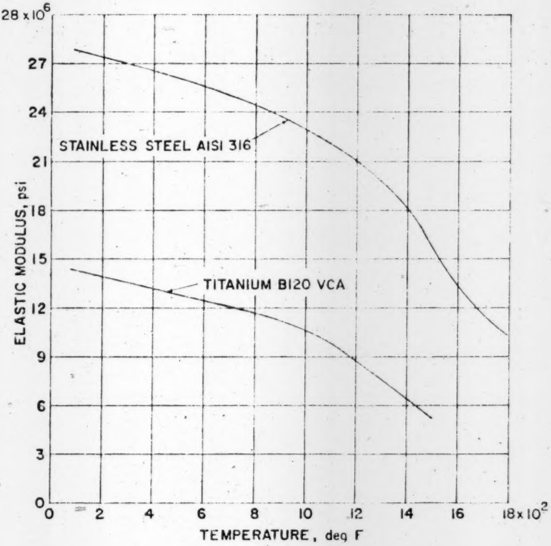


FIG. 18.—Tensile Modulus of Elasticity for B120-VCA Titanium Alloy and AISI Type 316 Stainless Steel for Hold Times to 1800 sec and a Strain Rate of 0.004 in. per in. per min.

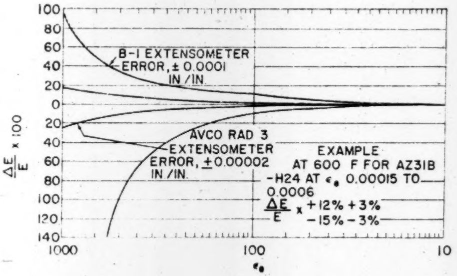


FIG. 19.—Maximum Error in the Modulus of Elasticity Versus Maximum Available Elastic Strain for ASTM Class B-1 and AVCO-Designed Extensometers.

With respect to point 3 above, each specimen was examined across the gage length to ensure that proper tolerances were maintained during machining. Specimens out of tolerance would be expected to exhibit excessive temperature gradients across the gage length because of area differences, which carries the discussion into point 4 above.

The results of the procedure outlined previously for the measurement of temperature gradients of the eight alloys tested indicated that these findings were in good agreement with those reported in reference (9) for similar specimens. It was found that the gradients along the lengths of aluminum bars varied within 10 F. Similar checks on the other materials indicated that the gradients were not excessive for the useful application of the data and that specimen development and heating techniques should be continued to minimize these gradients. The more extensive study of temperature measurement and gradients on Haynes No. 25 is more fully discussed later.

Points 5, 6, and 7 above are the result of the human factor, undoubtedly the greatest contribution to error in the determination of the elastic modulus. The extensometer system itself has the capability of measuring the room-temperature moduli of most materials to well within an accuracy of ±1 per cent and to within ±3 per cent for curves at elevated temperatures having an elastic strain of greater than 0.0005 in. per in. Superimposed on this error is that of the operator in properly mounting and aligning the extensometer so that no slippage will occur during the test (point 5); in carefully controlling the strain rate to maintain early stage creep in control, especially at elevated temperatures (point 6); and finally, in the interpretation of the stress-strain records (point 7) in which error is usually not greater than 3 per cent above the 0.0005 in. per in. strain level. Below this strain, the selection of a modulus line is arbitrary and the best-fit line is completely dependent upon the experience and judgment of the operator. Therefore the maximum cumulated human error in the determination of this property is assumed to range from 4 per cent at the room-temperature strain levels to about 6 per cent above the 0.005 in. per in. strain levels (approximately 600 F for 6061-T6 aluminum alloy). Differences as great as 25 to 30 per cent are possible for elastic strains less than 0.0005 in. per in., and at this point the judgment of the stress analyst (the user of the information) would provide a more realistic basis in the selection of stiffness criteria—for example, secant or tangent modulus.

TABLE 10.—THERMOCOUPLE REPRODUCIBILITY MEASUREMENTS.

Test Condition	Nominal Test Temperature, deg F				
	1000	1400	1600	2000	2300
B bar, without extensometer	1010 1016	1402 1403	1604 1604	1996 1996	2300 2293
C bar, without milled section	996 993	1403 1401	1604 1602	2000 1999	2305 2298
C bar, with milled section	996 995	1399 1400	1602 1603	1995 1998	2281 2290

Close tolerances were maintained in the machining of all specimens and fixtures to minimize the main causes producing nonaxial loading conditions. Also, the extensometer was designed as an averaging strain measuring instrument, thus minimizing the effect of smaller nonaxialities.

Extension of Useful Ranges of the Rapid Heating System:

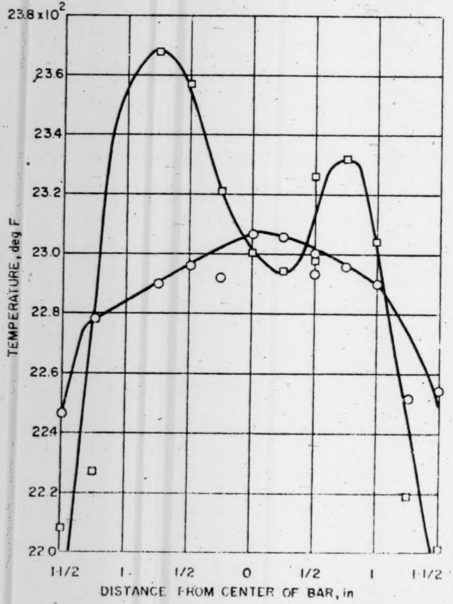
The performance of the chromel-alumel thermocouples was in general much better than had been expected. Readings of two thermocouples that were directly opposite each other on each of the bars are presented in Table 10 for three of the test conditions investigated. (Note that these readings were not expected to equal the nominal test temperature since they were not taken at the center of the gradient.) These results show that for short times, temperatures are reproducible to 3 or 4 F at 2000 F and to 8 or 9 F at 2300 F. The error begins to increase rapidly near 2300 F, which is due in part to contamination of the thermocouple by carbon when the insulation burns. These figures are well within the limits of error stated by the manufacturer of ±0.75 per cent between 530 and 2300 F, and were achieved by fabricating all thermocouples from a single lot of wire and by exercising careful welding procedures. An even smaller range might be obtained in a testing program by the secondary calibration of each thermocouple used. It appears that chromel-alumel thermocouples will not be sufficient to meet the accuracy demands of future test programs in the range above 2000 F and, of course, cannot be used to 3000 F. For this purpose, platinum, platinum-rhodium thermocouples should be considered for temperatures up to 2875 F, and for even higher temperatures tungsten alloy thermocouples or a pyrometer may be used.

Typical gradients obtained in this investigation are presented in Figs. 20 to 23. The exact shape of the gradient depends considerably on the method of holding each end of the specimen, the thermal conductivity of the material (3), the number of thermocouples and method of fastening (4), the position of the extensometer (5), and the amount of radiation from the surface. Severe longitudinal gradients have been experienced by several investigators. Southern Research Inst. (44) found gradients at the outer $\frac{1}{2}$-in. extremities of the 2-in. gage length of somewhat different specimens of various materials to be as much as 100 to 200 F at 1700 F. Typical gradients obtained in this investigation at the highest test temperature, 2300 F, are presented in Fig. 20 for the first two conditions considered and in Fig. 21 for the other two. These figures show the effects of the extensometer and the reduced section on gradients at any one temperature. Change in gradients with temperature is illustrated in Fig. 22 for the third condition. The change in gradient shown in this figure is identical for the fourth condition, and the same general pattern is also followed by the other conditions.

It had been expected that thermal gradients would increase rapidly at the higher temperatures of this study. Such is the case if the extensometer is attached; however, if it is not attached it was found that the gradients remain more or less constant, as can be seen in Fig. 22. These and other findings are listed below:

1. Without an extensometer, magnitudes of gradients show little change with temperature. This means that percentagewise the effect of gradients becomes less as temperature increases.

2. With the extensometer, gradients increase as expected. The shape of the gradient is shown in Fig. 20 having two distinct maxima. This shape of gradient was not found in exploratory tests with 6061 aluminum. It is believed to be caused by heat, generated due to the resistance of the knife-edges when they sink into the softened bar at high temperature, which cannot be transferred away by the lower conductivity L605 alloy. The two peaks begin forming slowly at about 1600 F and increase more rapidly above 2000 F. It is instructive to note that NACA (9) did obtain this type of gradient with aluminum. Temperatures outside the 1-in. gage length of their specimens similar in



□ with extensometer; ○ without extensometer

FIG. 20.—Thermal Gradients at 2300 F Nominal Test Temperature Along a 3-in. Reduced Section of a 12-in. L605 Test Bar.

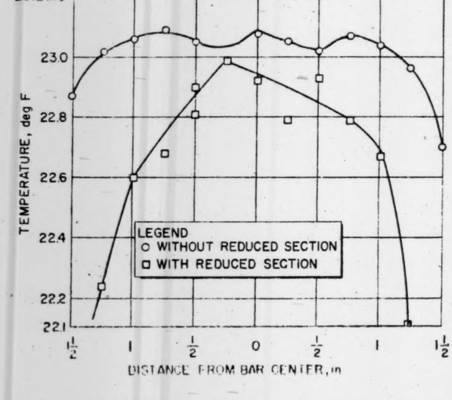


FIG. 21.—Thermal Gradients Along a Rapid-Heating-Rate Type C Specimen at 2300 F Nominal Temperature.

design to those used in the previous work were 20 to 30 F higher at 500 F than those inside the gage length.

3. Gradients in the C specimen were more severe with the milled central section than without, as shown in Fig. 21. This effect becomes noticeable between 1000 and 1400 F.

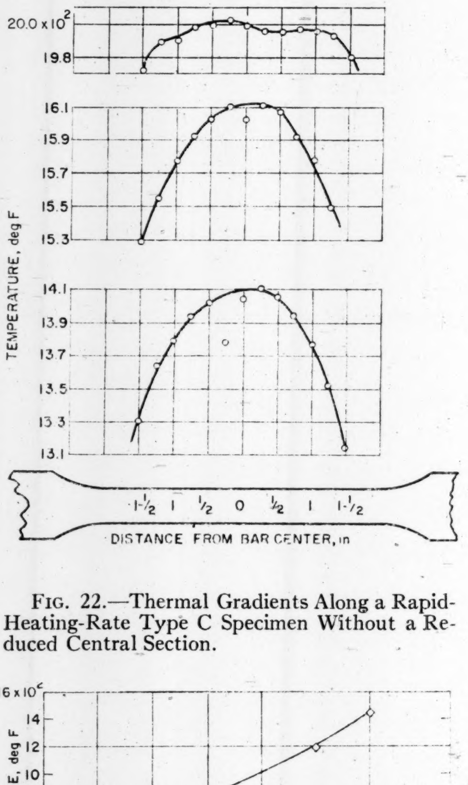


FIG. 22.—Thermal Gradients Along a Rapid-Heating-Rate Type C Specimen Without a Reduced Central Section.

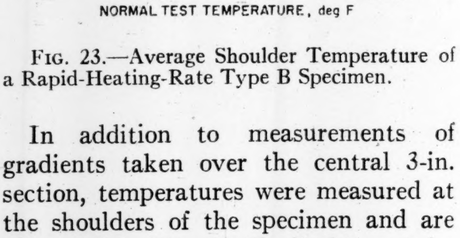


FIG. 23.—Average Shoulder Temperature of a Rapid-Heating-Rate Type B Specimen.

In addition to measurements of gradients taken over the central 3-in. section, temperatures were measured at the shoulders of the specimen and are presented in Fig. 23. Radiation, particularly for large specimens, may lower the surface temperature below that of the interior, thus creating transverse thermal gradients. This effect becomes important at very high temperatures. Such gradients were investigated by Southern Research Inst. (**44**) and were found to be an order of magnitude of 15 F at 1200 F. The differences in readings obtained in the present investigation between the sandwiched and outer thermocouples of one pair, mounted as described earlier, are presented in Table 11.

TABLE 11.—TRANSVERSE GRADIENTS OF SANDWICH SPECIMEN.

Nominal temperature, deg F	900	1100	1300	1500
Transverse gradient, deg F	13	13	14	22

TABLE 12.—EXTENSOMETER OPERATING TEMPERATURES.

Test Condition	Nominal Test Temperature, deg F		
	1600	2000	2300
Single-point tooth	956	1289	1534
Double-point tooth	967	1318	1585
Radiation shields, facing specimens	120 129	160 176	188 204
Strain gage, facing specimen	129 131	175 177	210 212
Strain gages, back to specimen	127 134	181 147	201 215

Above 1500 F, the thermocouples sandwiched in the center of the specimen failed, but extrapolation of the data given above tentatively indicates that the gradient at 2300 F would be at least 60 F. It is noted that these measurements were taken from a sandwiched specimen whose thickness is twice as great as that of an individual bar; hence, the transverse gradients in a type B or C bar are assumed to be about one half of those given in the table. No attempt has been made to adjust the temperature measurements taken in this work with the data in the table to arrive at an average temperature of the cross-section. Further investigation of this problem is highly desirable.

A problem that had not been anticipated at these higher temperatures was failure of the material of which the extensometer is constructed. The arms of the rapid-heating-rate extensometer, as previously described, are constructed of Micalex 400, a resin-reinforced ceramic. In the first series of tests, this material softened and distorted after having been in the temperature range of 1600 to 2300 F for about 45 min. As this instrument is to be used in future work up to 3000 F, other materials are recommended for investigation for use as arms.

In order to determine temperature levels for use in the redesign of this instrument, thermocouples were placed at 12 locations on the extensometer. The more important of these measurements are presented in Table 12.

The table shows that, within this temperature range, the temperatures reached by the various portions of the extensometer vary linearly with the nominal test temperature. Since strain gages are recommended by the manufacturer for service up to 400 F, no problems are anticipated with these gages until testing is conducted at well over 4000 F, provided this linear relationship holds. However, some difficulty has been experienced with the bonding agent above 300 F, and this may become a problem at 3000 F test temperatures.

The design of a similar rapid-heating-rate extensometer with a 1-in. gage length instead of the present 2 in. is recommended. Such a reduced gage length would reduce the longitudinal gradient problem.

Haynes No. 25 is reported to be resistant to oxidation to 1900 F. Observations of the behavior of the test bars show that the oxidation appears similar to high-temperature atmospheric corrosion of other cobalt alloys, such as Haynes Stellite. When heated in air, the alloys begin to tarnish on short hold time at 750 F and lose appreciable weight at 1380 F and above (1). A tightly adhering protective scale usually forms after the initial attack and prevents further oxidation. The oxide spalls on cooling due to the differential thermal expansion between the oxide coating and the base metal.

A bar that had been held at high temperatures for about an hour was sectioned metallographically at the point of contact of a thermocouple and showed that virtually no diffusion of the thermocouple into the base metal occurred. Measurements made on this specimen show that a layer of oxide as much as 0.005 in. may be lost through spalling from each face. The oxide layer that remains despite spalling is quite thin, the depth ranging from 0.0002 to 0.0008 in. The effect of spalling in reducing the effective cross-section of the specimen may be important but is unknown at present.

SUMMARY

The rapid-heating-rate unit developed for the purpose of simulating reentry heating conditions on metallic structural materials has been successfully applied in the determination of engineering data on eight different alloys. The data clearly indicate the extension of the useful temperature ranges of various alloys for very short-time engineering applications.

It is feasible to continue the development of the rapid-heating-rate facility to include the testing of refractory alloys up to their melting points for the determination of engineering properties. Inert atmospheres and vacuum chambers may be conveniently incorporated into the present system for such a program.

This facility provides a very useful and flexible research tool for performing high-temperature studies involving basic time-dependent metallurgical processes and their relationships with stress strain and time, all of which may be projected into practical use in missile designs.

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EFFECT OF OXIDATION ON STABILITY OF THERMOCOUPLES

BY C. D. STARR[1] AND T. P. WANG[1]

SYNOPSIS

The stability of emf of a thermocouple is shown to be independent of uniform oxidation but dependent on preferential oxidation. By using the alloy curves for formulating thermocouple alloys, the nature of the change in emf can be approximated. Comparisons are made for a type K thermocouple.

A thermocouple changes its electromotive force (emf) when used at elevated temperatures. The rate at which it changes can depend on the time, temperature, environment, composition of the alloys, wire diameter, and perhaps the metallurgical structure *per se*. While a substantial amount of qualitative information on the stability of thermocouples has been accumulated over the years, the amount of quantitative information, by contrast, is quite limited. One reason for this is that no standard or accepted method for stability testing is recognized. Consequently most tests are designed to simulate conditions that the thermocouple will see in service. But if new thermocouples are to be developed that will possess better stability than existing ones, a more fundamental understanding of stability is required. The objective of this paper is to attempt to show semiquantitatively the cause for instability of emf and to test this hypothesis against existing data.

GENERATION OF THERMAL EMF

The thermal emf of a thermoelement is dependent on its chemical composition. As with most properties, the first solute addition to a base metal has the largest effect on the thermal emf. Thus by adding sufficient amounts of solute to a base metal, a point can be reached where minor variations in the composition will not significantly alter the emf. This is necessary in order to be able consistently to manufacture alloys with a reproducible emf. It is also desirable from a stability point of view since minor changes in composition during use will not materially alter the emf. However, all existing conventional thermoelements made from base metals, except OFHC copper, are not simple metals or alloys. In order to obtain a more uniform thermoelectric power and to assure formability, a number of alloying additions are used, but care is used in their selection so that minor variations can be tolerated and interactions between alloying additions are minimized.

STABILITY OF EMF

The emf of an alloy can be altered by changing its chemical composition. This can occur either by the addition of an element during unintentional alloying—contamination—or by the removal of an element by preferential oxidation or volatilization. This paper will consider only the aspect of oxidation on the stability of thermocouples.

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ELECTRICAL ANALOG OF THERMAL EMF

In a simple thermoelectric circuit, the positive and negative thermoelements are connected in series. When the emf is measured with a potentiometer, the current flow in the circuit is zero when the galvanometer is at balance and no voltage drop occurs. The measured emf, therefore, is simply the algebraic difference of the emf's of the positive and negative thermoelements. However, when a thermoelement becomes oxidized, a simple series circuit is no longer obtained but instead a more complex one that must at least contain the components shown in Fig. 1. In this diagram it is assumed there is no interaction between the oxide and metal and the components act as though they are in parallel. The symbols E_p, E_o, and E_n represent the thermal emf's of the positive thermoelement, oxide, and negative thermoelement (which can be platinum) respectively while R and I refer to the resistance and current. When the emf of this assembly is measured with a potentiometer, the current from the parallel positive thermoelement and oxide, I_{po}, is zero at balance. However, at balance, a loop current still flows in the upper branch of the circuit so the emf is not merely the algebraic difference of the positive and negative thermoelements but instead

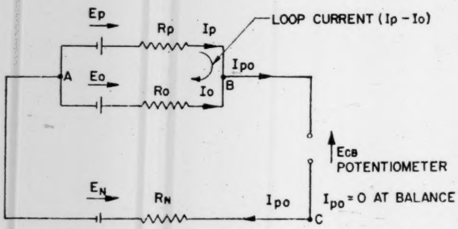

FIG. 1.—Circuit Diagram of an Electrically Parallel System in a Potentiometer Circuit.

$$E_{CB} = E_p - (E_p - E_o)\frac{R_p}{R_p + R_o} - E_n \quad \text{. . (1)}$$

where:

E_{CB} = emf of a circuit containing one oxidized thermoelement in series with a reference thermoelement,

R_p = resistance of unoxidized core of positive thermoelement, and

R_o = resistance of oxidized surface sheath of the positive thermoelement.

For minor amounts of oxidation, R_o approaches infinity and the second term reduces to zero, yielding the conventional emf, $E_p - E_n$. If testing is carried to destruction by completely oxidizing the thermoelement at elevated temperatures, the emf can be easily deduced by rearranging Eq 1 as follows:

$$E_{CB} = E_p\left(\frac{R_o}{R_p + R_o}\right) + E_o\left(1 - \frac{R_o}{R_p + R_o}\right) - E_n \quad \text{. . (2)}$$

Since R_p approaches infinity in this case, the emf reduces to $E_o - E_n$.

The gross features, but not the details, of this model have been demonstrated experimentally (**1**).[2] When very little or no oxide forms as in initial calibration, Δ emf $= E_p - E_n$. When a test is carried to destruction as McElroy (**1**) did, the emf change, prior to the resistance changing appreciably, is small. But as the resistance increased to several thousand ohms, corresponding to conversion of the metallic thermoelement to the oxide, an appreciable change occurred. Here the Δ emf approached the value $E_o - E_n$. Between these two extremes, the change in emf is real and finite and depends on the nature of the oxidation process.

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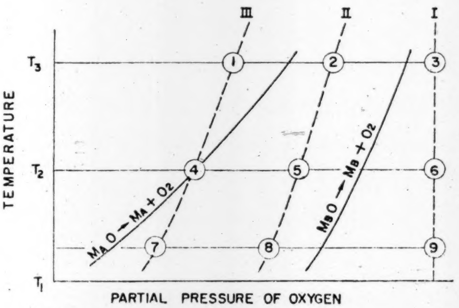
[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

Uniform Oxidation:



TEMPERATURE	COMPONENT	ATMOSPHERE I	II	III
T_3	A	O	O	R
	B	O	R	R
T_2	A	O	O	N
	B	O	R	R
T_1	A	O	O	O
	B	O	R	R

O = OXIDATION R = REDUCTION N = NEUTRAL

Fig. 2.—Effect of Oxygen Pressure on the Preferential Oxidation of *A* or *B* from Alloy *AB*.

Uniform oxidation is defined as oxidation that occurs without altering the composition or homogeneity of a thermoelement. In this case the thermoelement will be uniformly oxidized at some elevated temperature leaving a core of diameter d_p that has the identical composition of the initial alloy. The change in emf will, therefore, be a function of only the properties of the initial alloy and the oxide formed. Since the change in emf is the difference in emf between the thermoelement after testing and its initial value, the change is given by the second term in Eq 1. This term can be converted with the aid of the definition of resistance to

$$\Delta\text{emf} = (E_p - E_o)\left\{\frac{1}{1 + \frac{\rho_o}{\rho_p}\cdot\frac{d_p^2}{d_w^2 - d_p^2}}\right\} \quad ..(3)$$

where:

ρ_o = resistivity of oxide in ohm-cm,

ρ_p = resistivity of alloy in ohm-cm,

d_w = initial diameter of wire, cm, and

d_p = diameter of core, cm.

For a positive type K thermoelement (91Ni-9Cr) compared to platinum at 1000 C, E_p = 33 mv and $\rho_p = 136 \times 10^{-6}$ ohm-cm. The oxide formed will have a resistivity of about 1.5×10^2 ohm-cm and an emf between +227 and −175 mv depending on its composition (**1**,**2**). For an 0.128-in. diameter wire (d_w) that has *uniformly* oxidized until a solid core of 0.060-in. diameter remains, the Δ emf calculated is only ±1 μv which corresponds to less than $\frac{1}{30}$ of a degree change. Thus uniform oxidation will not cause a significant change in emf.

Preferential Oxidation:

When an alloy is exposed to an atmosphere containing a partial pressure of oxygen at an elevated temperature, the constituents of the alloy will not oxidize unless the partial pressure equals or exceeds the equilibrium pressure of the oxide. This is shown schematically in Fig. 2 where the equilibrium curves for the oxidation of two elements *A* and *B* from a binary alloy *AB* are shown on a temperature - partial pressure of oxygen diagram. If the pressure of oxygen in the atmosphere is greater than the equilibrium pressure, equilibrium can only be achieved by consuming the oxygen. Thus an atmosphere having an oxygen pressure greater than the equilibrium value will be oxidizing. Similarly, if the atmosphere has less oxygen than the equilibrium value, dissociation of the oxide will have to occur to reach equilibrium and reduction occurs.

As shown in Fig. 2, if alloy *AB* is exposed to an atmosphere having a partial pressure of oxygen as indicated at I, the atmosphere will be oxidizing to both components *A* and *B* of the alloy irrespective of the temperature. This is the condition for all base metals and alloys that are exposed to an abundant supply of air.

For a partial pressure of oxygen indicated by II, the atmosphere will be oxidizing to component *A* but reducing to *B* irrespective of the temperature. This is one condition that causes preferential oxidation such as is encountered in the "green rot" of 91Ni-9Cr (positive type K thermoelement). In this case, component *A* is chromium and component *B* is nickel. The preferential oxidation causes internal oxidation of the chromium *in situ* to the green Cr_2O_3.

This condition occurs for a base metal or alloy in many atmospheres where a reaction between the components of the atmosphere yields a low partial pressure of oxygen. Typical combinations of gases are N_2, H_2, and H_2O; H_2 and H_2O; CO and CO_2; N_2, CO, and CO_2; or more complex atmospheres containing all of the gases listed above. In stagnant atmospheres where the oxygen supply cannot be readily replenished such as in a long protection tube having a small diameter bore, low oxygen pressure can also be obtained.

For the preferential oxidation of chromium from a 91Ni-9Cr alloy at 2000 F, the atmosphere becomes reducing to nickel in the alloy when the partial pressure of oxygen is reduced below about 10^{-9} atmospheres. However, the atmosphere does not become reducing to chromium until a partial pressure of oxygen of 10^{-16} atmospheres is reached. Thus in between these two limits, the atmosphere is oxidizing to chromium and reducing to nickel.

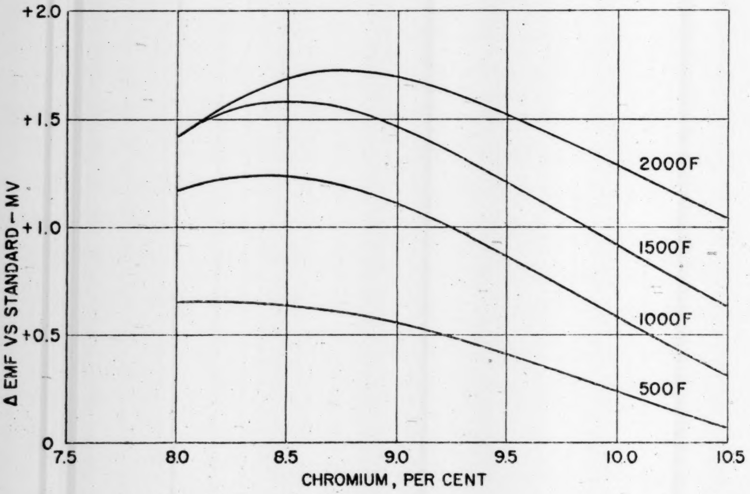


FIG. 3.—Effect of Chromium in a Nickel-Chromium (Tophel) Alloy on the emf at 500, 1000 1500, and 2000 F.

When the atmosphere produces a partial pressure of oxygen as indicated by III (Fig. 2), the conditions for oxidation are temperature-dependent. At a temperature of T_1, the atmosphere is reducing to *B* but oxidizing to *A*. At T_2, the atmosphere is neutral to *B* since the partial pressure of oxygen in the atmosphere is just equal to the equilibrium pressure of oxygen over the oxide M_BO. This atmosphere is of course reducing to *A*. At temperature T_3, the atmosphere is reducing to both *A* and *B*. As a result, the environ-

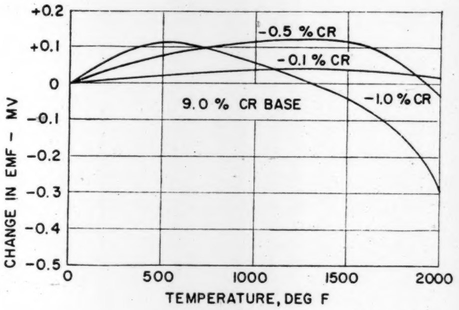


Fig. 4.—Change in emf of a Nickel Alloy with 9 Per Cent Chromium Caused by Preferential Oxidation of 0.1, 0.5, and 1.0 Per Cent Chromium Respectively.

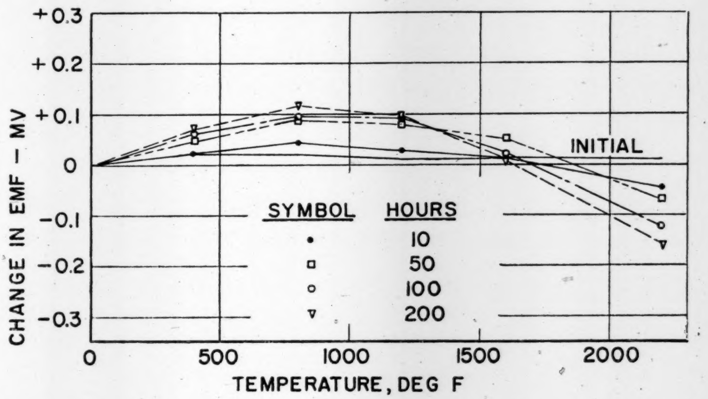


Fig. 5.—Changes in emf of Nickel-Chromium Alloy Due to Heating in Air at 2200 F for Times Indicated (Data from Dahl (6)).

ment *per se* can cause preferential oxidation. But preferential oxidation can occur by another method. The effect of oxygen pressure in Fig. 2 is based on thermodynamic data that are dependent only on equilibrium states and not on the rate at which equilibrium is achieved. Thus even though the atmosphere is oxidizing to both constituents, the rates at which they oxidize can be, and most generally are, different. Hickman (3) and Gulbransen (4) have shown by electron diffraction that the nature of the oxide formed on alloys similar to constantan and positive type K thermoelements are markedly different at different temperatures. This can only be caused by nonuniform oxidation.

In nonuniform oxidation, both the composition and the homogeneity of the thermoelement changes during the formation of an oxide. As shown previously, in the case of uniform oxidation the presence of the oxide alone does not cause a change in emf of the thermoelement. The change must, therefore, be attributable to the alteration in the composition of the core.

The general trend of the shift in emf during stability testing can, therefore, be deduced by determining the effect of loss of alloying constituents on the emf of a thermoelement. For this purpose, the data obtained in developing Tophel[3]-Nial[3] (type K thermocouple) are used to calculate the general shapes of the stability curves obtained by Dahl (5) many years ago.

Positive Type K Thermoelement (91 Ni-9 Cr)

Figure 3 shows the effect of chromium in nickel on the deviation in emf from an arbitrary standard at four temperatures. Note the maximum emf is not achieved at the same chromium content for all temperatures. For the base alloy, 9 per cent chromium, the Δ emf is approximately +0.55, +1.1, +1.45, and +1.7 mv at 500, 1000, 1500, and 2000 F respectively. When the alloy is exposed to a temperature such that 0.1 per cent chromium is preferentially oxidized, the remaining alloy has an effective composition of 8.9 per cent. The Δ emf of this composition can be read from the graph and the difference from the original 9 per cent alloy obtained. This is shown in Fig. 4 where the change in emf from the original 9 per cent alloy is shown against temperature for chromium losses of 0.1, 0.5, and 1.0 per cent chromium.

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[3] Trade name, Wilbur B. Driver Co.

At low temperatures where oxidation is slow, or at high temperatures for short times, a small loss in chromium will occur and the emf will shift in a positive direction. When more chromium is lost, the shift in the positive direction is larger at lower temperatures but becomes negative at high temperatures. As more chromium is lost, the positive deviation is shifted to lower temperatures and the deviation at high temperatures is more negative. This is in accordance with experimental data as shown in Fig. 5. A comparison of Figs. 4 and 5 suggests that about 0.75 per cent chromium was preferentially oxidized in the test conducted by Dahl (5).

When a type K thermocouple is exposed to an exothermic atmosphere such as 10 per cent CO, 5 per cent CO_2, 16 per cent H_2, 1 per cent H_2O, 1 per cent CH_4 and balance nitrogen commonly used in furnaces for brazing

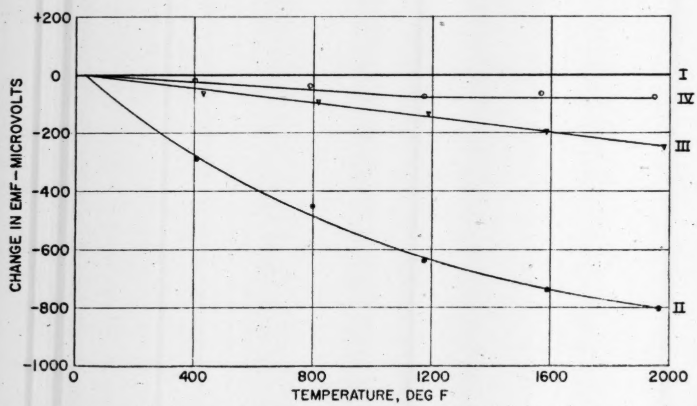


FIG. 6.—Effect of Temperature on the Change in emf of a Positive Type K Thermoelement After Various Treatments.

Curve I, reference curve; Curve II, after exposure to exothermic atmosphere; Curve III, after first grinding; Curve IV, after second grinding.

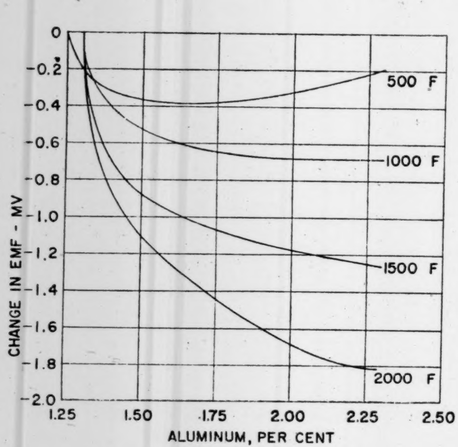


FIG. 7.—Effect of Aluminum on the Change in emf of a Nial Alloy at 500, 1000, 1500, and 2000 F.

copper, the oxygen content of the atmosphere corresponds to II in Fig. 2. Under this condition preferential oxidation of chromium occurs and the nickel-chromium leg becomes magnetic at room temperature after only a few hours, indicating that the chromium content has been reduced to less than 7 per cent.

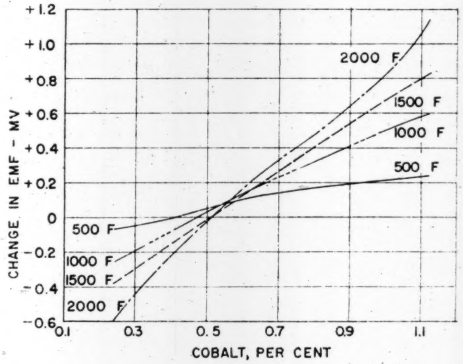


FIG. 8.—Effect of Cobalt on the Change in emf of a Nial Alloy at 500, 1000, 1500, and 2000 F.

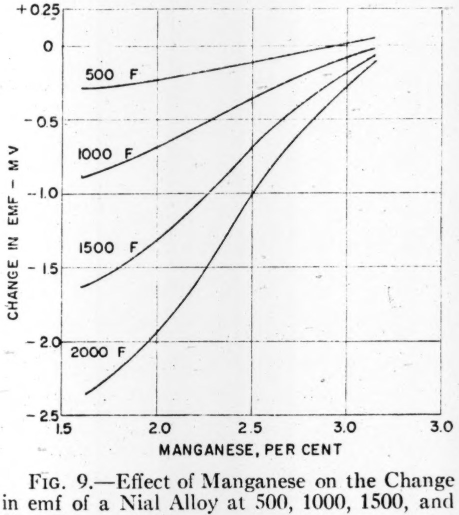


FIG. 9.—Effect of Manganese on the Change in emf of a Nial Alloy at 500, 1000, 1500, and 2000 F.

This type of reaction alters the simple thermocouple to one wherein the emf of the positive thermoelement is approximated by the form of Eq 1 where

$$E^1 = E_{9\mathrm{Cr}} - (E_{9\mathrm{Cr}} - E_{7\mathrm{Cr}}) \frac{R_{9\mathrm{Cr}}}{R_{9\mathrm{Cr}} + R_{7\mathrm{Cr}}} \quad (4)$$

in which E^1 = emf of type K thermoelement with a preferentially oxidized peripheral surface containing 7 per cent chromium in solid solution.

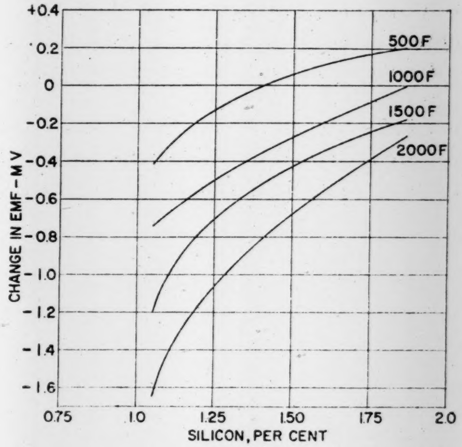


FIG. 10.—Effect of Silicon on the Change in emf of a Nial-Base Alloy at 500, 1000, 1500, and 2000 F.

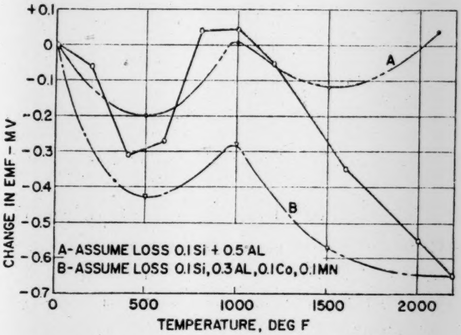
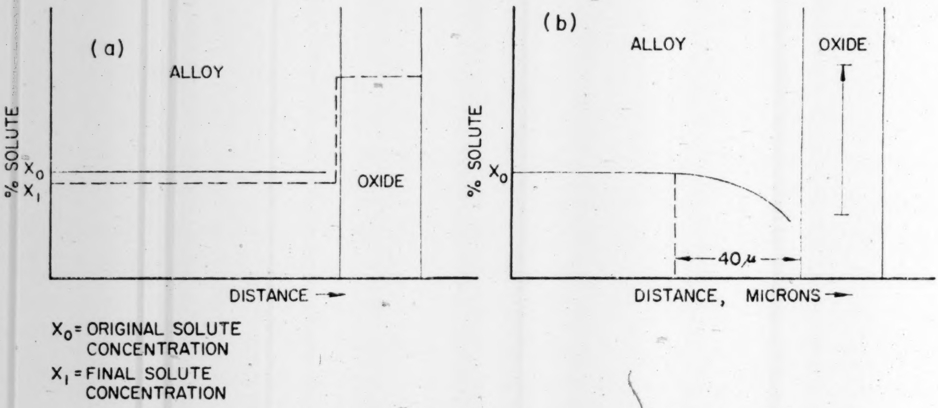


FIG. 11.—Comparison of Shape of Curve of Change in emf of Ni-Mn-Al-Co-Si Alloy Due to Heating in Air 200 hr at 2200 F with Calculated Curves (Experimental Curve from Data by Dahl (6)).

As shown in Eq 4, if preferential oxidation is nil the second term on the right-hand side of the equation is zero. But as preferential oxidation proceeds both E_{7Cr} and R_{7Cr} decrease, causing an increase in the last term. As a result, instead of a change of a few degrees in the positive direction, a large negative change in emf can occur. Values as high as −4 mv equivalent to −180 F at a test temperature of 1750 F have been noted.

Conversely, if the parallel path through the preferentially oxidized zone is eliminated, the original emf should be restored. To test this hypothesis, a positive type K thermoelement (91Ni-9Cr) was exposed to an exothermic atmosphere to cause preferential oxidation until the surface was magnetic. The emf referenced to its original value was then measured in accordance with procedures in ASTM Method E 207[4] and is shown in Fig. 6. By uniformly grinding the thermoelement to remove a portion of the oxidized zone and thereby decreasing the second term in Eq 4, the Δ emf should decrease. As shown in Fig. 6, this did occur. Finally, by removing essentially all of the preferentially oxidized zone the emf reverted to within 100 μv of its initial value, clearly showing the effect of preferential peripheral oxidation on the emf of the thermoelement.



(*a*) Solute distribution assuming homogeneous metal composition.
(*b*) Actual solute distribution.

FIG. 12.—Solute Distribution in Binary Alloy After Oxidation.

NEGATIVE TYPE K THERMOELEMENT, NI + 5 (MN, CO, AL, SI)

The negative thermoelement of the type K couple is a nickel-base alloy containing aluminum, cobalt, manganese, and silicon. Figures 7 to 10 show the effect of variations of the major alloying additions on the emf at 500, 1000, 1500, and 2000 F. By a process similar to that used in the simple binary system above, the general nature of drift can be deduced. The results for two assumed conditions are shown in Fig. 11 along with an experimental curve by Dahl (**5**). In case *A*, it was assumed that 0.1 per cent silicon and 0.5 per cent aluminium were preferentially oxidized and that no interaction occurred. In case *B*, it was assumed that silicon, aluminum, cobalt, and manganese were lost in the amounts noted. While neither assumption fits the experimental curve, the general trends are readily apparent. In each case, the trend in Δ emf is the same at 500, 1000, and 1500 but differs at 2000 F. While a more judicious choice of combination of the four elements could be made to duplicate more closely the experimental curve, nothing new would be demonstrated.

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[4] Method of Thermal EMF Test of Single Thermoelement Materials by Comparison with a Secondary Standard of Similar EMF-Temperature Properties (E 207), 1962 Supplement to Book of ASTM Standards, Part 3.

DISCUSSION

The change in emf during stability testing can be approximated by an estimation of the amount of preferential oxidation. Exact correlation cannot be obtained for a number of reasons. First the composition curves in Figs. 3, 7, 8, 9, and 10 were obtained during the development of Tophel-Nial while the stability data were taken from the literature. Since the composition of the thermocouples has changed over the years, exact correlation would not be anticipated. But the main source of error concerns the morphology of the specimens. In this paper it has been assumed that preferential oxidation occurs but that the temperature is high enough and the time long enough for diffusion to yield a homogeneous core up to the metal-oxide interface as shown in Fig. 12(*a*). In this schematic diagram, X_0 is the original solute concentration and X_1 the final solute concentration after the alloy has been oxidized.

However, it is well known that oxidation results in a nonhomogeneous matrix. Most investigations have generally used either microexaminations or hardness to show a change as the oxide-metal interface is approached (6–8). But within the past few years a new tool, the electron microprobe beam analyzer, has been made available which affords a method for quantitatively evaluating the chemical composition of the metallic zone contiguous to the oxide (9). Figure 12(*b*) shows an actual solute composition curve as determined by the microprobe beam analyzer. The distance of the depleted zone behind the oxide-metal interface will depend on time, temperature, and the alloy system. The composition of the oxide will not be uniform as indicated in Fig. 12(*a*) but will vary considerably as shown in Fig. 12(*b*). If the electrical resistance of this zone is high, the variation in composition will not be significant with respect to emf stability. However, the variation in composition of the matrix will be significant because the electrical resistance of successive cylindrical cores will be small and of the same order of magnitude but the emf will differ. As the cross-section is divided into smaller segments and the correct emf of each section used, a closer approximation can be expected than by assuming a homogeneous distribution of the solute elements.

For more complex alloys, a series of curves, one for each solute addition, is needed in Fig. 12(*b*). A good approximation for the change in emf should be obtained by treating each alloying element separately, since most alloys for thermocouples are formulated such that the interaction term between elements is small compared to the effect of the alloying element itself.

CONCLUSIONS

1. The oxide formed on base metal thermoelements does not contribute significantly to the change in emf until just prior to complete oxidation of the element.

2. Uniform oxidation of base metal thermoelements does not cause a significant change in emf.

3. Preferential oxidation of base metal thermoelements is the primary cause for emf instability because it changes the composition of the matrix. This type

of oxidation is caused either by the environment *per se* or by the rate at which the alloying elements in a thermoelement oxidize.

4. The general nature of the change in emf can be deduced by a knowledge of the elements preferentially oxidized and their effect on emf.

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(9) K. G. Carroll, private communications.

CELL TEST METHOD FOR STUDY OF ALKALI-CARBONATE ROCK REACTIVITY

By J. E. Gillott[1]

Synopsis

Argillaceous dolomitic limestone from Kingston, Ont., expands when placed in a strongly alkaline environment. Such expansion may disrupt concrete that contains aggregate made from this rock. A cell technique has been developed to test samples for reactivity. The apparatus is described and results are illustrated with reference to eight samples of concrete. Petrographic, mineralogical, and chemical analyses are given for the samples tested. The cell technique is discussed in relation to other test methods. It is possible to recognize potentially reactive carbonate rocks petrographically, but it is not possible to predict the amount of expansion a particular sample will produce. Linear measurements of concrete beams give reliable results, but the cell technique is more rapid. Dimensional change measurements of rock prisms soaked in alkali may give erroneous results if undetected microcracks develop. It is also difficult to allow for anisotropic properties of rocks, and many samples must be studied if the results are to be representative of a quarry. The cell technique eliminates these difficulties; powder is used and the expansive reaction may be smoothly followed to completion.

When argillaceous dolomitic limestone from Kingston, Ont., is used as aggregate it produces cracking and deterioration in concrete made with high-alkali cement. The concrete failure is caused by expansion of the rock in strong alkali. Similar behavior by aggregates has been reported from other localities (**1**,**2**).[2] Swenson (**3**) found that on the basis of test criteria developed for alkali-aggregate reaction up to the time of the initial investigation, the Kingston aggregate would not be considered deleteriously reactive. Concrete beams made with Kingston coarse aggregate cracked after several months' exposure to high humidity conditions and measurements showed that a systematic elongation occurred (**3**,**4**). Expansion was also measured on rock prisms soaked in alkali (**4**,**5**) and the method has been considered tentatively as a test method (**6**).

The concrete beam test, though reliable, is time consuming; measurements made on more than one rock prism soaked in alkali are needed if the information is to be representative, since great differences in reactivity are known within a single quarry. Directional properties in the rock such as bedding also influence results (**4**, Fig. 10). A test method has been developed in which powdered rock is used. The sample is readily made representative, variations due to directional properties are eliminated, and the fine particle size gives a rapid response. The apparatus is described and the behavior in this test of eight samples of

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

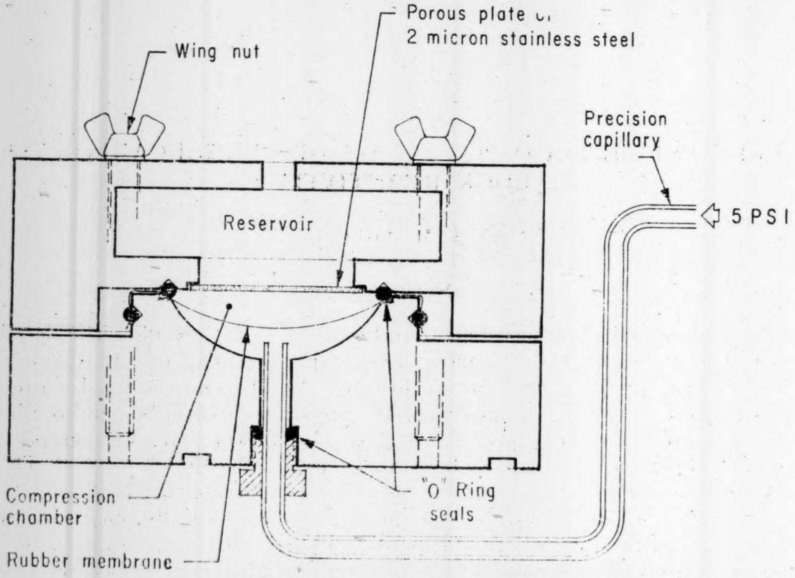
Wing nut
Porous plate of
2 micron stainless steel
Precision
capillary
5 PSI
Reservoir
Compression
chamber
Rubber membrane
"O" Ring
seals

FIG. 1.—Cell for Testing Reactive Aggregate.

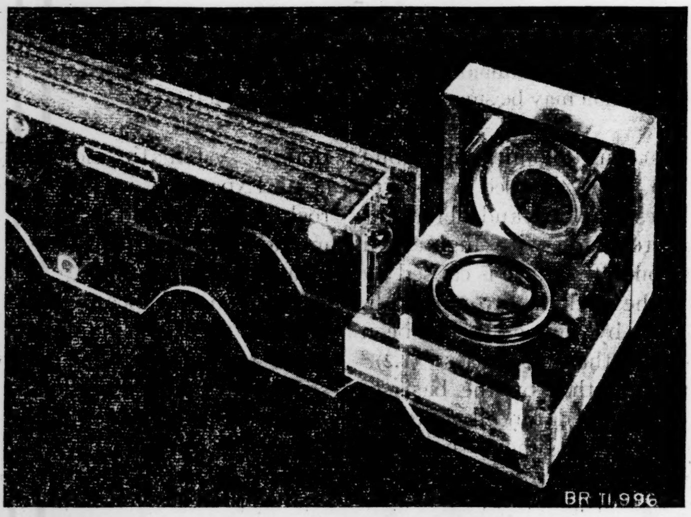

FIG. 2.—Two Halves of a Cell, Capillary Tube, and Metric Scale Used to Measure Volume Change in Powdered Aggregate.

known performance in concrete is discussed.

EXPERIMENTAL PROCEDURE

The apparatus is an adaptation of that used by Warkentin et al (7) in work on clays (Figs. 1 and 2). A powdered sample was mixed with a little water and placed on the rubber membrane in the compression chamber and sealed with O-rings. It had access to water or alkali via a porous plate of stainless steel of 2-μ pore diameter. The chamber below the rubber membrane was filled with water leading to the precision (1-mm bore) capillary tube so that volume changes in the compression chamber were registered by the meniscus. Phenolphthalein solution was added to this water so that any leakage of alkali through the rubber membrane would have been apparent. The sample was confined under a constant air pressure of 5 psi, and the whole apparatus was immersed in a water bath to maintain a constant temperature of 22 C.

TABLE 1.—DOLOMITE-CALCITE COMPOSITION AND REACTIVITY OF CARBONATE ROCKS.

Sample	Partial Analyses, per cent				Calculated				Total	Reactivity
	Acid-Insoluble	Fe	Ca	Mg	Dolomite,[a] per cent	Calcite, per cent	Calcite: Dolomite			
							Weight	*M*		
Kingston composite	5.69	0.46	28.34	6.38	48.42	44.50	0.92	1.69	99.07	Reactive
Virginia B	14.97[b]	0.70	24.68	5.85	44.36	37.53	0.85	1.56	99.31	Reactive
Georgia	5.31	0.34	30.85	4.94	37.44	56.70	1.51	2.79	99.45	Reactive
Indiana B	14.08	0.43	26.64	5.21	39.55	45.06	1.14	2.09	99.12	Reactive
Indiana A	6.18	0.28	33.27	2.99	22.66	70.76	3.12	5.77	99.60	Slightly reactive
Ohio	6.67	1.88	25.66	7.84	59.43	31.81	0.53	0.99	99.79	Slightly reactive
Virginia A	1.87	0.11	34.33	3.47	26.3	71.45	2.72	5.00	99.62	Unreactive
Alabama	0.57	0.13	33.59	4.58	34.72	65.03	1.87	3.45	100.32	Unreactive

[a] Calculated from magnesium and calcium assumed present only as carbonate.

[b] Plus 1.75 per cent aluminum dioxide.

At the start of each experiment the sample had access to deionized or distilled water in the reservoir via the porous plate. When a slowed contraction was registered by the meniscus, indicating little further compaction of the powder under a pressure of 5 psi, the water was replaced by a 2 *M* solution of alkali.

In the initial experiments the solution was circulated continuously by a peristaltic pump. Similar results were obtained, however, when the solution was left static in a reservoir behind the porous plate, and a reservoir was incorporated in the design of the apparatus illustrated. Experiments showed that about 100 ml of alkali were required to exhaust the expansive capabilities of 30 g of rock. The expansive reaction normally went to completion in about two weeks, and the whole experiment took about one month.

DESCRIPTION OF SAMPLES

Rocks of different petrographic character, ranging in composition from almost pure dolomite to limestone, were obtained. Some of the samples were known to be reactive and were supplied by other workers. All were subjected to either the concrete beam test or the rock prism test. Those rocks that expanded in the tests were used in investigating the reliability of the cell technique as a test method. Certain of the samples produced no expansion in the rock prism test and concrete beam test but were still used in the cell test. Partial chemical analyses of the samples described here are given in Table 1, where the percentage composition is shown in terms of acid-insolubles, dolomite, and calcite. The carbonates were calculated from the experimentally derived values for magnesium and calcium on the assumption that these elements were present only as carbonates. The value shown for the calcite-to-dolomite ratio by weight and by moles was also calculated. These samples all fall within the composition fields of dolomitic limestone or calcitic dolomite (8), which includes the critical range of composition for alkali-carbonate rock reaction. Within this range, however, there seems to be no simple relationship between composition and reactivity. The very slightly reactive sample from Ohio, for example, is not much different in its percentage of acid-insoluble or carbonate composition from the highly reactive Kingston material. The calcite-to-dolomite ratio of the unreactive Alabama rock is not much different from the reactive Georgia sample, though there is a much lower content of acid-insoluble material. This is also true of the unreactive rock from Virginia and of other nonexpansive samples where the carbonate composition suggested potential reactivity.

TABLE 2.—COMPOSITION OF CARBONATES IN TERMS OF MOLAR PER CENT.

Sample	Dolomite[a]		Calcite[a]		Reactivity
Kingston:					
6 to 7 ft bed	$Ca_{55.6}Mg_{44.4}$	± 0.72	$Ca_{99.53}Mg_{0.47}$	± 0.21	Reactive
10½ to 12 ft bed	$Ca_{55.51}Mg_{44.49}$	± 0.77	$Ca_{97.90}Mg_{2.10}$	± 0.71	Reactive
20 to 21 ft bed	$Ca_{55.14}Mg_{44.86}$	± 0.65	$Ca_{96.17}Mg_{3.83}$	± 0.94	Reactive
Virginia B	$Ca_{54.51}Mg_{45.49}$	± 0.49	$Ca_{98.48}Mg_{1.52}$	± 0.44	Reactive
Georgia	$Ca_{55.08}Mg_{44.92}$	± 0.79	$Ca_{98.12}Mg_{1.88}$	± 0.43	Reactive
Indiana B	$Ca_{52.40}Mg_{47.60}$	± 0.68	$Ca_{98.75}Mg_{1.25}$	± 0.46	Reactive
Indiana A	$Ca_{51.65}Mg_{48.35}$	± 2.01	$Ca_{98.10}Mg_{1.90}$	± 0.47	Slightly reactive
Ohio	$Ca_{53.48}Mg_{46.52}$	± 1.22	$Ca_{98.54}Mg_{1.46}$	± 0.73	Slightly reactive
Virginia A	$Ca_{54.74}Mg_{45.26}$	± 0.68	$Ca_{98.70}Mg_{1.30}$	± 0.24	Unreactive
Alabama	$Ca_{50.92}Mg_{49.08}$	± 0.10	$Ca_{99.34}Mg_{0.66}$	± 0.27	Unreactive

[a] ± Tolerance gives the 95 per cent confidence interval for the mean value shown for either element expressed in mole per cent.

Goldsmith and Graf (9) showed that dolomite may contain an excess of calcium carbonate above the theoretical 50 *M* per cent and that the lattice of such a dolomite is commonly disordered and the structure metastable. Such a dolomite is described as nonideal. Calcite sometimes shows variation in composition also, the most common being a replacement of calcium by magnesium. These changes of composition lead to such characteristic effects on the X-ray powder diffraction pattern as a line shift resulting from a change in lattice spacing. This may be determined by accurate measurement of X-ray powder patterns and is related to the composition of the dolomite or calcite expressed as *M* per cent magnesium carbonate in calcium carbonate. The composition of the dolomite and calcite in the rocks described in this paper was determined in this way from X-ray films taken with a Guinier quadruple focusing camera (Table 2). Results for the Kingston samples have been previously published (10). There is little sign of a correlation between composition and reactivity. Dolomite and calcite, which make up the unreactive Alabama rock, are both nearly ideal, whereas the unreactive Virginia A sample contains carbonates, which are very close in composition to the nonideal dolomite and slightly magnesian calcite of the reactive rocks.

The rocks were examined microscopically and some of the less familiar petrographic terms used are defined here:

1. *Intraclasts.*—Fragments of semiconsolidated sea-bed redeposited after local erosion.
2. *Micrite.*—Fine calcite ($CaCO_3$) in crystals of 1 to 4 μ in diameter.
3. *Intramicrite.*—A rock in which the dominant fragmental material consists of intraclasts in a matrix of micrite.

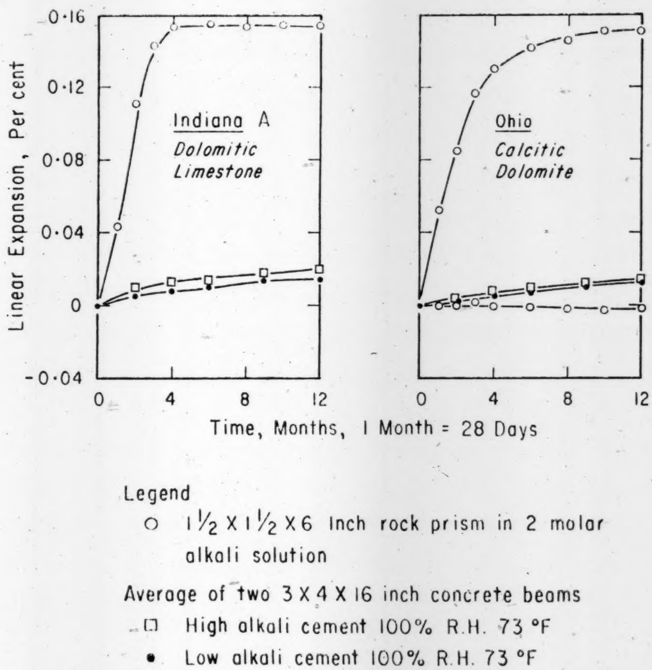

FIG. 3.—Expansion of Rock Prisms and Concrete Beams.

4. *Intrasparite.*—As No. 3, but matrix consists of clear calcite crystals generally larger than 10 μ.

More complete definitions are given in the literature (**11,12**).

Petrographic examination of the reactive Kingston rocks shows that they contain a high proportion of unrecrystallized fine lime mud or micrite (**11,12**). Bands occur of slightly disturbed calcareous ooze that contain a sprinkling of silt-size quartz. The dolomite euhedra attain a size of about 0.05 mm, but despite the extensive dolomitization, the fabric criteria of Bathurst (**13**) show that the matrix has not undergone grain growth or recrystallization. The rocks were deposited under generally quiet sedimentary conditions and have been dolomitized but not recrystallized (**10**). The petrography of the reactive rocks from Indiana, Georgia, and Virginia is quite similar to that of the Kingston rocks, although the dolomite euhedra reach a size of 0.2 mm in the Virginia B sample and 0.1 mm in the Georgia rock. Virginia B also differs from the Kingston samples in that skeletal material is present. The slightly reactive Indiana A sample contains beds of different microfacies. The rock prism, which expanded when placed in alkali (Fig. 3), is a dolomitized fossiliferous intramicrite, rather

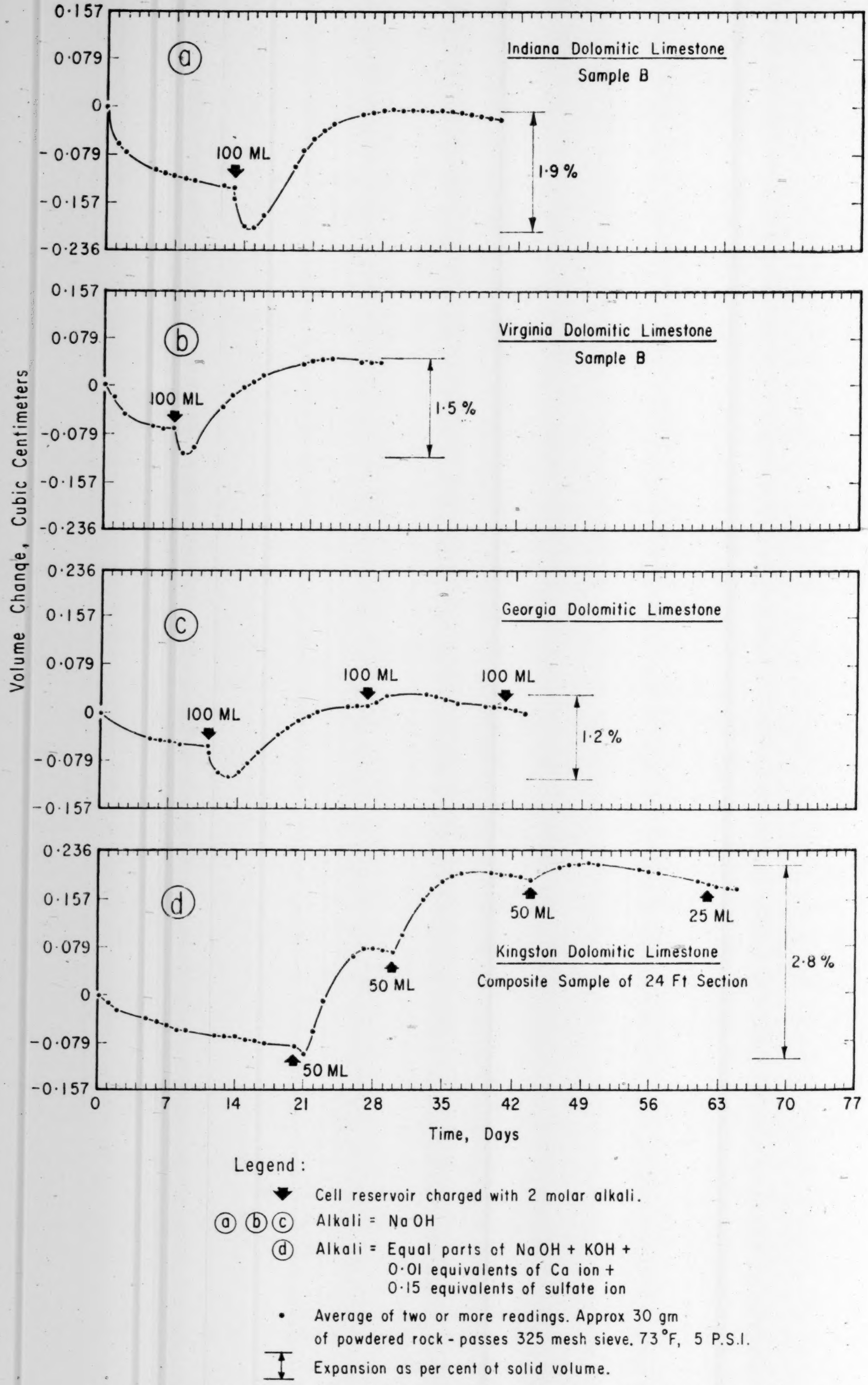

FIG. 4.—Expansion in 2 *M* Alkali of Reactive Carbonate Rocks in Cells.

similar to the Kingston rocks apart from the skeletal material. The remainder of these rocks contain more intrasparite and fossil fragments than the Kingston rocks. In addition to the biogenic material, some bands contain abundant oolites which are sometimes compacted. The conditions at the time of sedimentation of some, if not all, of these strata were probably different from those under which the reactive Kingston beds accumulated, since oolites are believed to indicate deposition in agitated water (14).

The rocks from Ohio and Alabama differ from the Kingston samples in that they are coarser grained, and crystals of both calcite and dolomite reach a size of about 0.2 mm or larger. Primary structures have been eliminated by recrystallization in places, but as this is of variable degree, intraclasts have elsewhere survived. These are now made up of 8 to 10-μ microspar and are surrounded by the coarser crystals of the matrix. A sample of Ohio rock that expanded in alkali (Fig. 3) shows less recrystallization than most of this material and retains a finely crystalline matrix. The unreactive Virginia A rock is an extensively dolomitized intrasparite, which is of coarser grain size than the Kingston material. The rock also has a cleaner appearance, due in part no doubt to the low acid-insoluble content.

EXPERIMENTAL RESULTS

The cell test was carried out first on a composite sample obtained from the top 24 ft of a quarry near Kingston and on a bed 6 to 7 ft from the surface. A sample of about 30 g of rock ground to pass a No. 325 sieve was used. As shown in Fig. 4(*d*), the composite rock from the quarry near Kingston expanded stepwise when 50-ml charges of 2 *M* alkali were supplied to the sample. The third addition, however, produced only slight further expansion, and the final addition none; the expansive capabilities of 30 g of rock were exhausted by the addition of a total of about 100 ml of 2 *M* alkali. In subsequent experiments a smooth expansion curve was obtained as 100 ml of 2 *M* alkali was used to replace distilled water in the cell reservoir as soon as equilibrium was attained.

The density of the powdered rock was determined by means of a pycnometer. As the weight of powder in the cell was known, the initial solid volume was obtained. The increase in volume was calculated from the experimental results obtained by use of the cell and hence the expansion as a percentage of the initial solid volume was calculated.

In the experiment in which the curve in Fig. 4(*d*) was plotted, the expansion in terms of the volume of solid amounted to about 2.8 per cent and the alkali was a solution of sodium, potassium and calcium hydroxide, and gypsum (4). In a later experiment in which pure sodium hydroxide was used, an expansion of nearly 8 per cent was registered. It is probable that this greater increase in volume was caused in part by the fact that the influence of the sodium hydroxide was more aggressive than that of the potassium hydroxide (4). Pure sodium hydroxide was used in the other cell tests described in this paper.

When distilled water was replaced by alkali, a sharp contraction was registered by the meniscus in the capillary tube, and expansion did not commence until two or three days after the alkali had been added to the system. The cause of this contraction is not definitely known, but further compaction of the powder may have resulted from the addition of alkali to the system, owing to a migration of water from between the particles of powder into the reservoir while alkali ions moved in the opposite direction via the porous plate. An alternative explanation is that while the sample was compacting

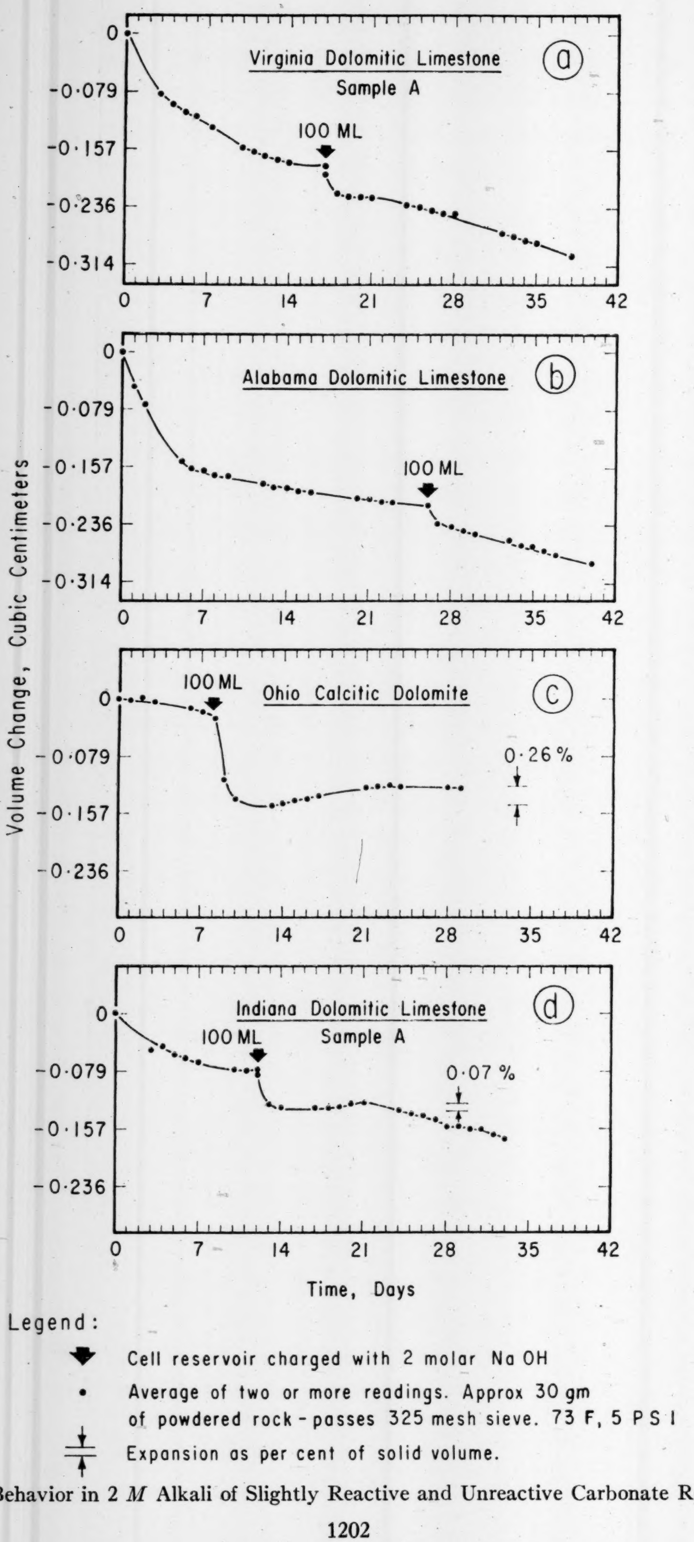

FIG. 5.—Behavior in 2 *M* Alkali of Slightly Reactive and Unreactive Carbonate Rocks in Cell.

in the presence of water in the cell reservoir a structure developed between the particles of powder, which ceased to be stable in the presence of alkali. This type of behavior is common in clay systems and is exploited commercially in ceramics where similar effects are induced by the addition of very dilute solutions of sodium carbonate. For the cell, it was found that the addition of 1 ml of 2 *M* sodium hydroxide to the distilled water in the reservoir resulted in a sharp contraction (registered by the meniscus in the capillary tube) similar to that following replacement of the water by 2 *M* alkali. Experience with nonexpansive samples showed that the rapid contraction normally lasted about two to three days, after which the curve assumed a slope approximately parallel to that of the curve prior to the addition of alkali (Figs. 5(*a*) and (*b*)). It seems probable that during the two days following the addition of alkali to reactive samples, this contraction prevented the registration of expansion by the meniscus in the capillary tube. If so, the delayed expansion does not imply a period of induction (the elapse of a time interval significant to the mechanism), before the expansion reaction commenced.

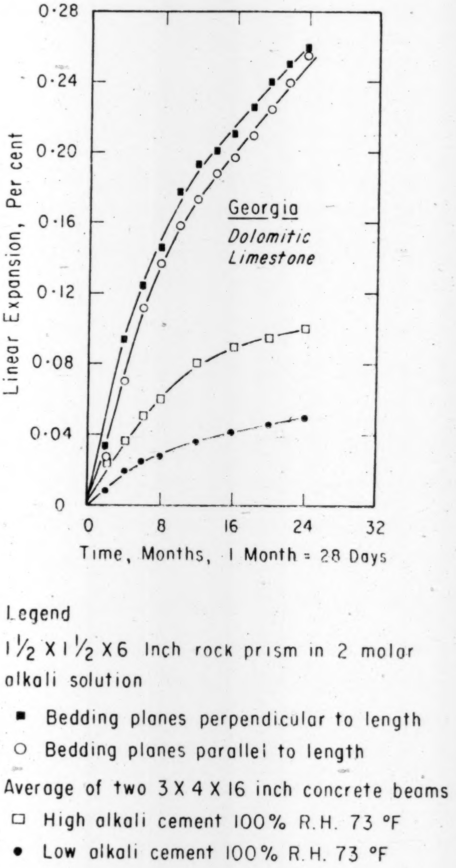

FIG. 6.—Expansion of Rock Prisms and Concrete Beams.

It was noticed also that after expansion had ceased the curve often had a greater downward slope than during the period of equilibrium prior to the addition of alkali. This may have resulted from an osmotic flow of water through the rubber membrane into the alkali, with a consequent contraction registered by the meniscus. Alkali was apparently unable to migrate in the opposite direction by this mechanism, since the water below the membrane remained neutral. The osmotic flow of water would, however, tend to counteract registration of expansion, which resulted in an apparently smaller volume increase than actually occurred. The curve obtained when using nonexpansive samples shows that the effect is of minor significance.

The profile registered during expansion is a fairly typical rate curve in which a high early expansion rate tapers off asymptotically with time, toward parallelism with the portion of the graph prior to the addition of alkali to the system (Fig. 4).

The linear expansion produced after two years by a reactive aggregate from Georgia when used in concrete beams made with high- and low-alkali cement and by rock prisms immersed in 2 *M* alkali is shown in Fig. 6. A split of 30 g of this rock, ground to pass a No. 325 sieve, produced an expansion of about 1.2 per cent in the cell test (Fig. (4(*c*)). A sample of reactive dolomitic limestone from Virginia and another sample from Indiana each gave a somewhat similar curve to the Georgia sample when used in a cell (Figs. 4(*a*) and (*b*)). Insufficient material was available from Virginia and Indiana for the concrete beam test, but the rock is known to cause expansion if used as aggregate[3] or in the rock prism test.[4]

Differences in response to the rock prism test have occurred in some cases. A sample from Ohio behaved in this manner: two prisms of the rock were immersed in alkali (Fig. 3), and while one produced a linear expansion of about 0.15 per cent at 10 months, the other showed none. Concrete beams made with high-alkali cement with this rock as aggregate showed only 0.02 per cent linear expansion after 2 yr exposure to high humidity conditions. A representative split of this rock, ground to pass a No. 325 sieve, produced only 0.26 per cent expansion in the cell test (Fig. 5(*c*)). Another example of this sort of behavior occurred with a sample from Indiana. Used as aggregate, this material caused a linear expansion of only 0.02 per cent in concrete beams, but a prism of the rock expanded 0.16 per cent in 4 months when immersed in alkali (Fig. 3). A representative sample of the material, when powdered, gave only 0.07 per cent increase of volume in the cell test (Fig. 5(*d*)). Hand specimens of Indiana rocks known to show differences of per cent linear expansion in the rock prism test showed fairly good agreement between the cell test and the rock prism test in that the rock producing the greatest expansion in the prism test showed the greatest volume increase in the cell test.

These and other results indicate that rock from different parts of the same quarry may show marked differences in behavior when placed in a strongly alkaline environment. It is therefore most important that the sample tested should be representative of the rock to be used as aggregate. This may be readily achieved for the cell test by quartering powdered rock or passing it through a splitter. Reliable sampling is much more difficult in any test method that relies on individual rock samples.

During these investigations over 50 carbonate rocks covering the composition range from limestone to dolomite were tested for expansivity by the concrete beam test and the rock prism test. Most samples produced no unusual expansion, even though some of them were similar in carbonate composition to the reactive rocks. A selection of unreactive samples was used in the cell test. None of those tested caused a volume increase, and typical profiles plotted during experiments with two of them are shown in Figs. 5(*a*) and (*b*).

CONCLUSION

By petrographic examination it is possible to recognize rocks similar to those from Kingston and elsewhere that expand deleteriously in alkali. A partial chemical analysis is also of some help since the calcite-to-dolomite ratio may be calculated and the acid-insoluble content determined. Since this falls between about 5 and 20 per cent in many of the reactive rocks it may well be significant to the mechanism. It is not possible, however, on the basis of these techniques, to predict the amount or degree of expansion that will be caused if rocks of this type are used as aggregate.

Alkali-carbonate rock reactivity may, however, be detected by the measurement of length change of concrete beams held under controlled conditions of 100 per cent relative humidity and 73 F.

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[3] H. Newlon, personal communication.

[4] D. W. Hadley, personal communication.

The advantages of this test method are that aggregate of the same size and composition may be used with the cement to be used in practice. Interaction between aggregate and cement occurs under conditions commonly encountered under natural exposure. The main disadvantage is that a conditioned room is required and it takes considerable time before definite conclusions can be drawn as to whether or not deleterious expansion will be caused by a particular aggregate.

Comparative linear dimensional change measurement of rock prisms soaked in alkali is another technique used to evaluate reactivity. By this means the rock may be shown to be the causative agent for the expansion found in concrete, but as a test method for aggregate it has several disadvantages. In particular many specimens must be studied to evaluate properly the reactivity of a representative sample of the rocks in a quarry. There is also an inherent danger of spurious results, since expansion is known to cause cracking, and microcracks may go undetected and cause errors in the evaluation of per cent linear expansion. In addition, the measurement of length change is less appropriate than volume change measurement to the understanding of behavior of concrete aggregate, because interaction with cement paste is three dimensional. Rocks also have strong directional properties such as bedding planes, joints, and cleavages, and results are likely to be strongly affected (**4**, Fig. 10) depending on the angle such planar structures make with the direction of linear measurement used to evaluate the dimensional change.

The cell test appears to provide a rapid and reliable means of estimating the reactivity in alkali of carbonate rocks. Since powder is used the sample is easily made representative of the rock to be used as aggregate. The small particle size of the powder ensures a rapid response to alkaline attack, and the increase of volume may be followed to completion with no danger of spurious results owing to cracking or anisotropy.

Acknowledgments:

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REPEATED-LOAD TRIAXIAL TEST OF SOILS WITH PORE PRESSURE MEASUREMENT

By Rodney W. Johnson[1] and Eldon J. Yoder[2]

A research program on the physical characteristics of sand-soil mixtures under repeated dynamic loads made by R. W. Johnson in 1957 required rather complex equipment that was not available commercially. This paper describes the equipment designed and constructed as a part of the research program as well as the methods and procedures developed to accompany the testing of certain soil specimens included in the program.

Dynamic Triaxial Loading Equipment

The research program consisted of the following distinct phases: (1) equipment design and construction, (2) development of testing techniques and procedures, and (3) testing program and data analysis.

It was determined that a repetitive dynamic triaxial-loading apparatus was needed that would also permit measurement of pore pressures in the sample. The equipment was designed by the authors and fabricated in the Civil Engineering shop of Purdue University.

A schematic diagram of the dynamic-loading equipment used for the triaxial tests is shown in Fig. 1. This equipment is based generally on an earlier, successful design used by Havers (**1**)[3] in his study of subgrade pumping. The loading is provided by compressed air actuating a piston-type air cylinder. The magnitude of the load carried to the soil sample by the piston can be controlled by regulating the air-line pressure. Compressed air is supplied by a 3-hp air compressor operating at 120 to 160 psi outlet pressure. From the air compressor the air passes into an accumulator tank of 0.9 cu ft capacity and through a combination 40-μ filter, pressure regulator, and oiler. As indicated in Fig. 1, the line branches to the dual-loading units which can be operated either singly or in unison. The system, designed for maximum flexibility of loading and calibrating operations, can be made to operate either single or double acting under dynamic loading; or maintain a continuous, static load. The desired dynamic or static-loading condition is controlled by the manually operated air-line valves and the four-way Bellows-Valvair pilot-operated solenoid valves (Fig. 2).

To energize the system, the line switch is closed to maintain current through the normally closed contacts of the switch in the counter to the normally closed contacts in the timer. This impulse energizes the solenoid valve, causing the port, which is normally closed, to open for the down or pressure stroke of the piston. After the controlled delay in the timer has elapsed, the contacts in the counter open, causing one count to be recorded and deenergizing the timer which, in

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[2] Professor of Highway Engineering and Research Engineer, Joint Highway Research Project, Purdue University, Lafayette, Ind.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper.

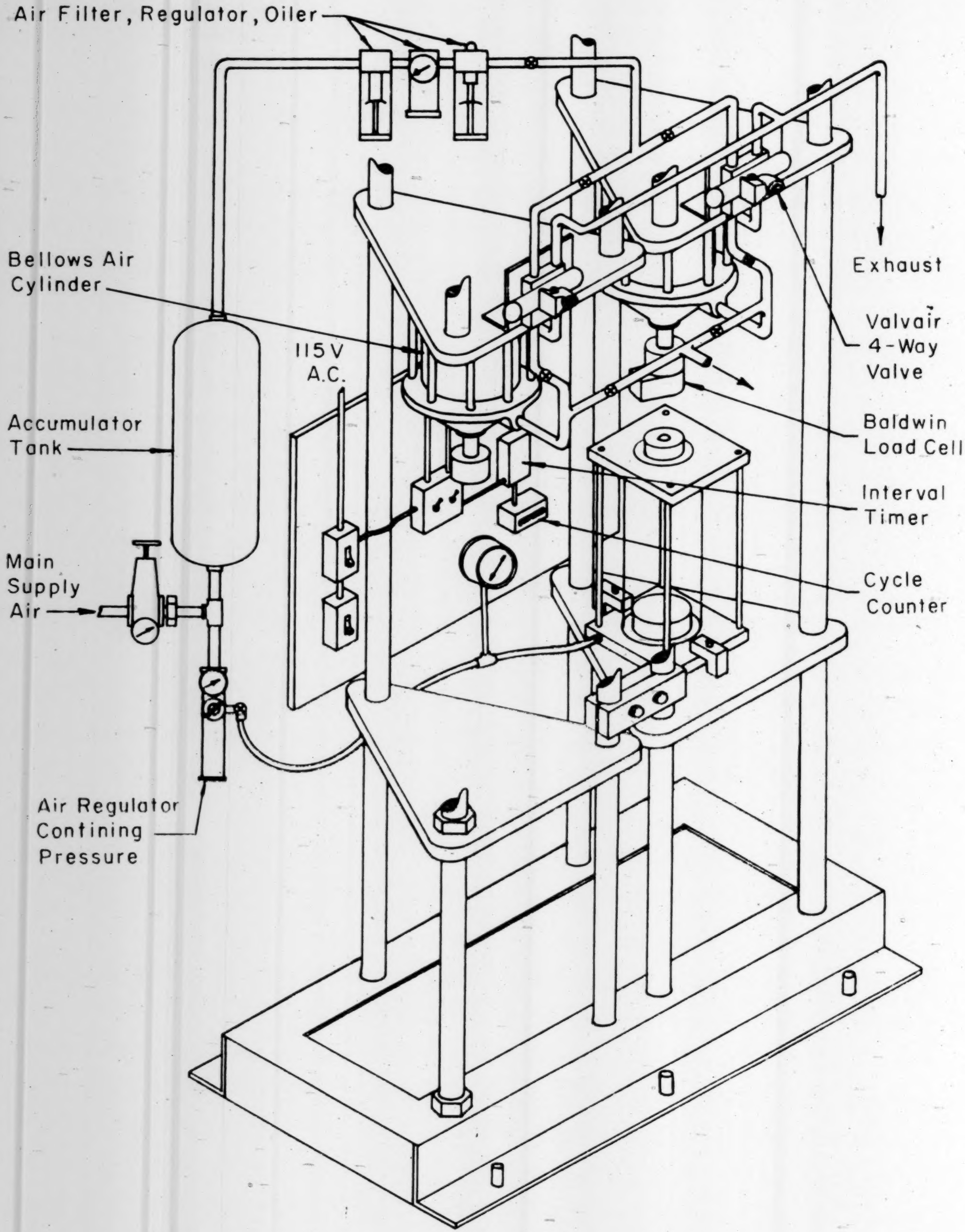

Air Filter, Regulator, Oiler
Bellows Air Cylinder
115 V A.C.
Exhaust
Valvair 4-Way Valve
Accumulator Tank
Baldwin Load Cell
Interval Timer
Main Supply Air
Cycle Counter
Air Regulator Confining Pressure

FIG. 1.—Dynamic Loading Equipment Used for Triaxial Tests.

turn, deenergizes the pressure stroke of the piston. The timer controls the duration of load and the interval between load applications for any desired number of repetitions as set on the counter. An adjustable microswitch set on the loading piston of the triaxial cell will shut off the entire apparatus after the

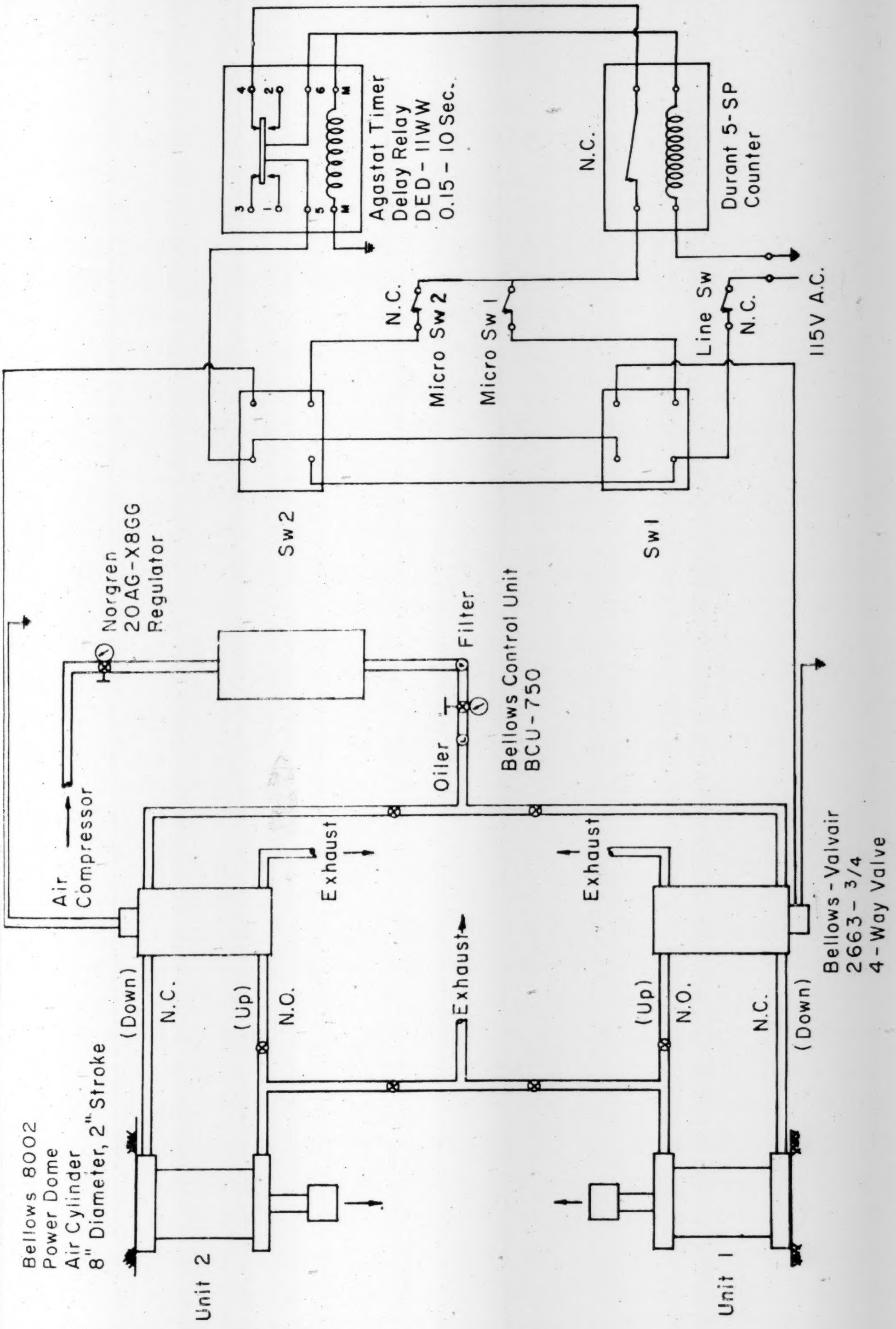

Bellows 8002
Power Dome
Air Cylinder
8" Diameter, 2" Stroke
Unit 2
Unit 1
(Down)
N.C.
(Up)
N.O.
Exhaust
Air Compressor
Norgren 20AG-X8GG Regulator
Oiler
Filter
Bellows Control Unit BCU-750
Bellows-Valvair 2663-3/4 4-Way Valve
Sw 2
Sw 1
Micro Sw 2
Micro Sw 1
Line Sw
N.C.
115V A.C.
Agastat Timer Delay Relay DED-11WW 0.15-10 Sec.
Durant 5-SP Counter
1
2
3
4
5
6
M

FIG. 2.—Air and Electrical Distribution System.

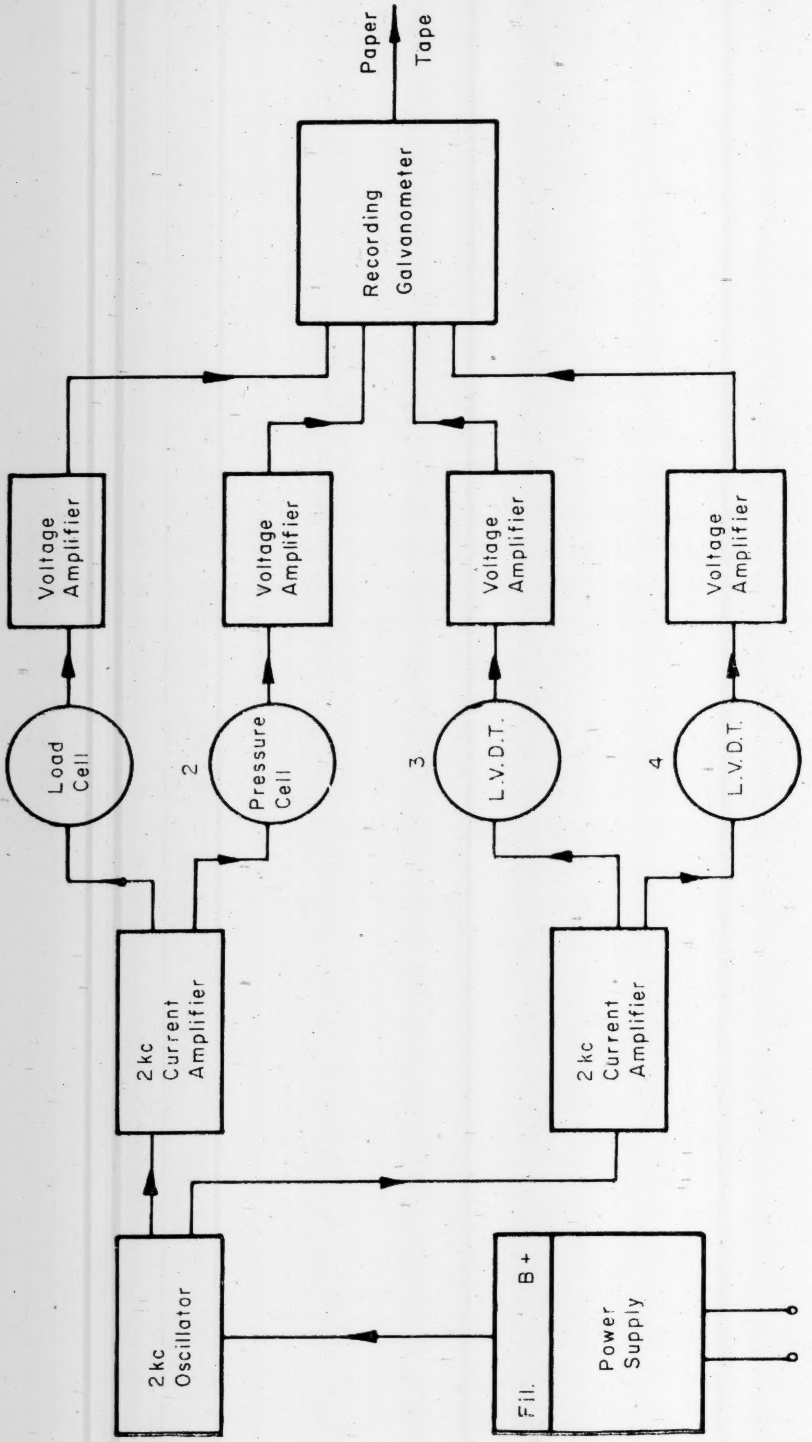

Paper
Tape
Recording Galvanometer
Voltage Amplifier
Voltage Amplifier
Voltage Amplifier
Voltage Amplifier
1
Load Cell
2
Pressure Cell
3
L.V.D.T.
4
L.V.D.T.
2kc Current Amplifier
2kc Current Amplifier
2kc Oscillator
Fil.
B+
Power Supply
115V A.C.

FIG. 3.—Components of Electronic Sensing and Recording Equipment.

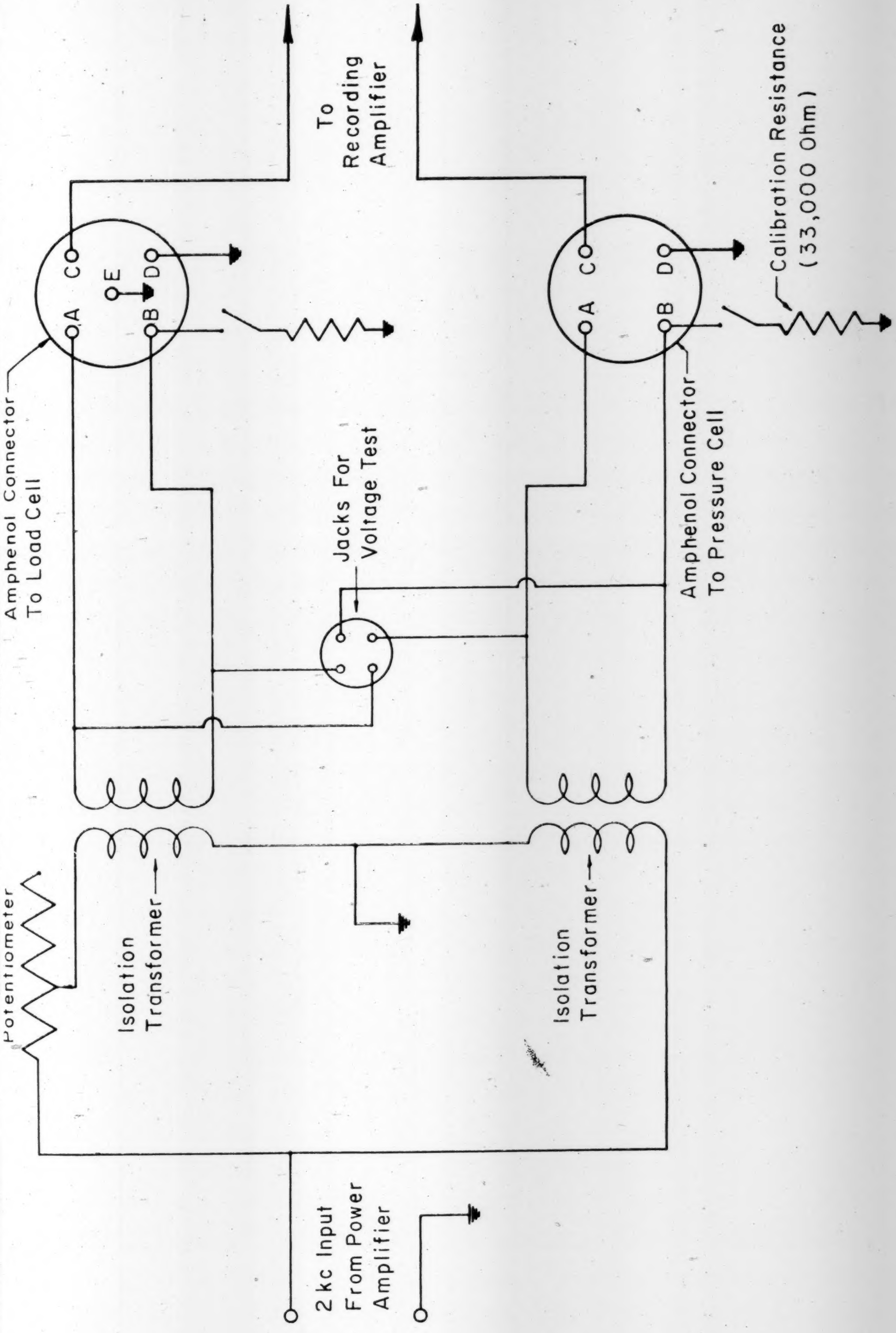

Amphenol Connector
To Load Cell
A
C
E
B
D
To
Recording
Amplifier
Jacks For
Voltage Test
Amphenol Connector
To Pressure Cell
A
C
B
D
Calibration Resistance
(33,000 Ohm)
Potentiometer
Isolation
Transformer
Isolation
Transformer
2 kc Input
From Power
Amplifier

FIG. 4.—Strain-Gage Bridge Unit.

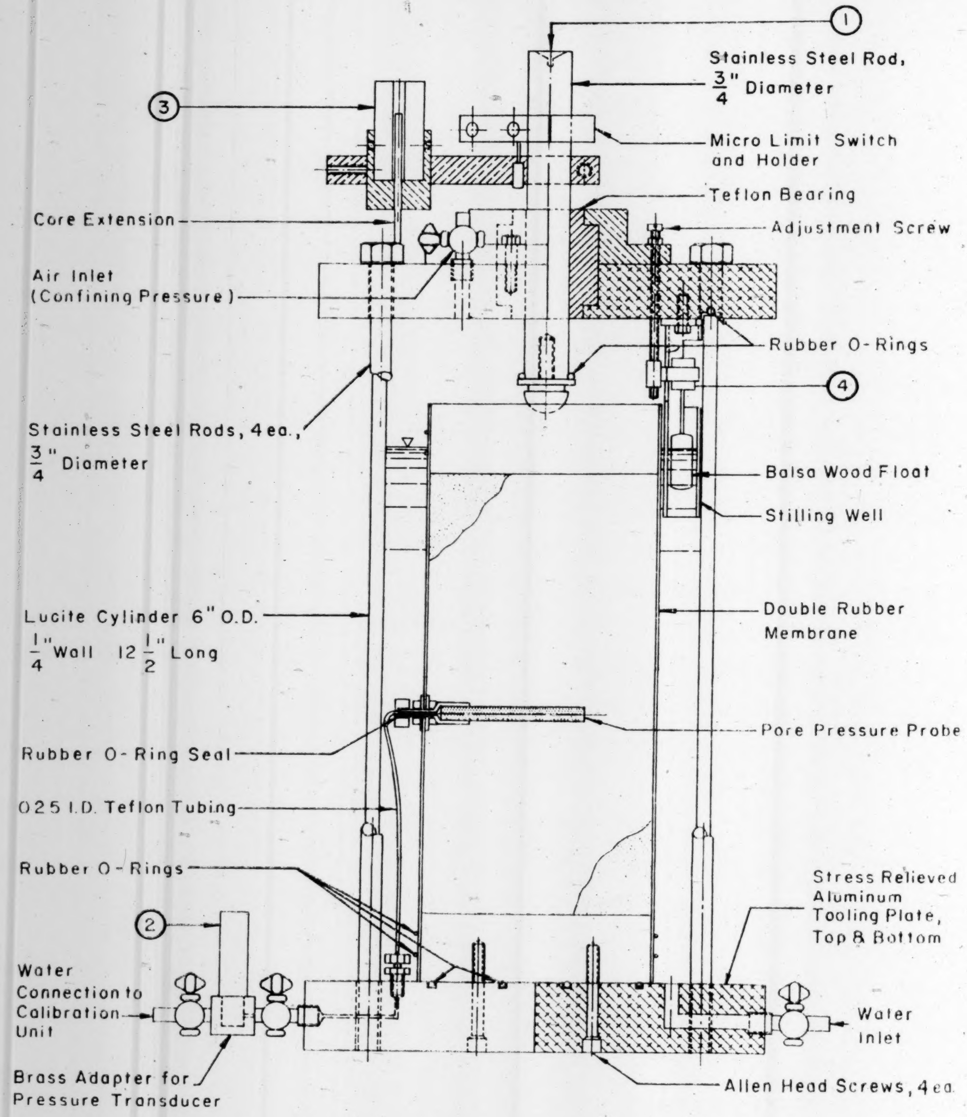

①
Stainless Steel Rod, $\frac{3}{4}$" Diameter
③
Micro Limit Switch and Holder
Teflon Bearing
Core Extension
Adjustment Screw
Air Inlet (Confining Pressure)
Rubber O-Rings
④
Stainless Steel Rods, 4 ea., $\frac{3}{4}$" Diameter
Balsa Wood Float
Stilling Well
Lucite Cylinder 6" O.D. $\frac{1}{4}$" Wall 12 $\frac{1}{2}$" Long
Double Rubber Membrane
Pore Pressure Probe
Rubber O-Ring Seal
025 I.D. Teflon Tubing
Rubber O-Rings
Stress Relieved Aluminum Tooling Plate, Top & Bottom
②
Water Connection to Calibration Unit
Water Inlet
Brass Adapter for Pressure Transducer
Allen Head Screws, 4 ea.

FIG. 5.—Triaxial Cell Showing Electronic Pickup Devices.

sample fails or after the deformation reaches a pre-determined magnitude. The foregoing sequence takes place in a single-acting movement in which it is desired to keep the loading piston in continuous and complete contact with the soil sample. For operation as a double-acting system, the manual control valves are opened or closed as necessary to obtain the desired action; no change in the timing sequence is required.

CONTROLLED TEMPERATURE ROOM

Accurate determination of pore pressures and volume changes, which are temperature dependent, requires that the testing be performed in a temperature-controlled environment. This was achieved by use of a controlled temperature chamber in which the loading apparatus and electronic gear was installed. In addition to the obvious advantage obtained by use of the chamber for fluid measurements, a secondary, though important, benefit was that of a stabilized temperature for all electronic sensing and recording elements, thus improving the performance and reliability.

A firm anchored support for the loading apparatus entirely free of contact with the chamber was achieved by carrying the weight of the apparatus through the floor of the chamber on six anchor rods. These anchors are not in contact with the chamber at any point and thus avoid extraneous disturbances by personnel in the room.

ELECTRONIC RECORDING EQUIPMENT

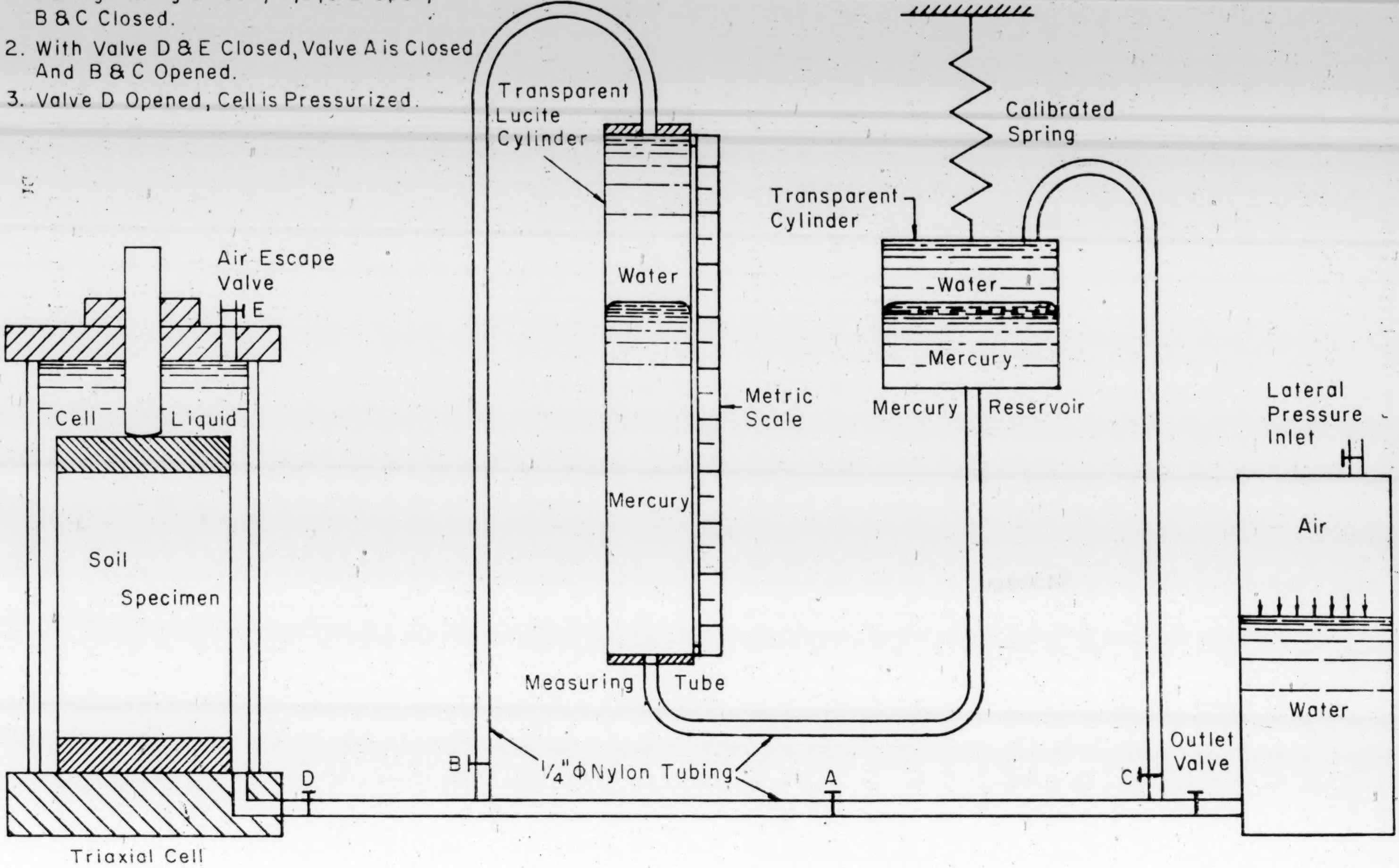
The electronic recording equipment used in this study consisted mainly of modified components designed and used on an earlier investigation of subgrade-support characteristics (2). A block diagram of the components is shown in Fig. 3 and the bridge and calibration unit in Fig. 4. The sensing elements, numerically keyed in Fig. 5, are shown in relationship to the test specimen.

The system is composed of a power supply unit, a seven-channel amplifying and control unit, a six-channel recording galvanometer unit, and various electronic pick-up devices. The amplifying and control unit contains seven amplifiers, seven frequency modulating units, seven power amplifiers for driving galvanometers, oscillators generating voltages at 30,000 cps and 2000 cps, and two power amplifiers. The amplifying unit was modified by removing one amplifier and inserting another power amplifier to provide additional energy for the sensing elements. A bridge and calibration unit (Fig. 5) was designed and constructed to permit use of strain gages since its original design was specifically for use with Schaevitz 040L and 125L linear-variable transformers. The bridge unit was inserted in the position formerly occupied by the top-most amplifier. The rectified 30-kc voltage is filtered and used as a bias for the power amplifier; the 2-kc voltage is amplified by the two-power amplifiers that serve as the energy source for the transducers.

The galvanometer unit consists of six BL-902 Brush Electronics Co. pen motors (input impedance 1450 ohms) and one event marker. All channels may be used with either ink-writing or electric-writing pens on sensitized paper. In this study the electric stylus was used. All galvanometer units are mounted in a single unit, Brush Model BL-276.

SENSING ELEMENTS AND CALIBRATION

A Baldwin Type C, 0 to 5000-lb load cell was used to measure the magnitude of the applied load including its own weight and that of the moving piston. The load cell, using four active SR-4 strain gages wired in a Wheatstone-bridge circuit, has a precision of ±0.25 per cent, full-scale calibration accuracy. Calibration of the load cell was accomplished by loading the cell in the environmental chamber against an accurate proving ring. Galvanometer and proving-ring deflections were recorded for varying magnitudes of applied load. The amplifier gain was brought into proper adjustment and the galvanometer pen zeroed. Simultaneously, the power amplifier output voltage was adjusted to operating voltage. Recordings were taken for several cycles of load and unload, and the mean of the values taken as the most probable value. With the cell completely unloaded and the dead load of


Transparent
Lucite
Cylinder
Calibrated
Spring
Transparent
Cylinder
Air Escape
Valve
E
Water
Water
Mercury
Cell
Liquid
Metric
Scale
Mercury
Reservoir
Lateral
Pressure
Inlet
Air
Soil
Specimen
Measuring
Tube
Water
B
$1/4$" Φ Nylon Tubing
A
C
Outlet
Valve
D
Triaxial Cell

NOTE:

1. During Filling Of Cell, A, D, & E Open; B & C Closed.
2. With Valve D & E Closed, Valve A is Closed And B & C Opened.
3. Valve D Opened, Cell is Pressurized.

FIG. 6.—Apparatus for Measuring Volume Change Under Pressure.

the piston removed, the calibration resistance was switched on to note and record the galvanometer-pen deflection.

From the work of Whitman et al (3), it was determined that measurement of the pore pressure using a Dynisco PT-25, 0 to 100 psia pressure transducer (Fig. 5) was possible. The cell uses a miniature strain gage in the configuration of a four-active-arm Wheatstone bridge. Performance characteristics include a maximum nonlinearity of 0.5 per cent full-scale and a repeatability maximum of 0.1 per cent full-scale.

Calibration of the pressure cell was accomplished in much the same manner as the load cell. A closed pressure chamber was constructed of about 1-liter capacity. The transducer adapter was threaded into the base, and the chamber was half filled with de-aired water. The pressure cell was then screwed into the adapter, its base being in and below water so that no air bubbles were present in the system. Air was admitted to the top of the chamber, and its pressure recorded on an accurate, laboratory-pressure gage. As the pressure was varied, galvanometer deflections were recorded for each increment of pressure change for several cycles of loading and unloading; the mean of these values was taken as the most probable value. Power amplifier-output voltage and calibration-resistance deflection were also noted and recorded; a high degree of linearity was obtained for both of these devices, particularly at the middle and upper ranges of the calibration curve.

Deformation was measured by a Schaevitz Type 125SL linear-variable transformer, that was mounted in an adjustable holder which, in turn, was mounted in a bracket attached to the loading ram of the triaxial cell. The transformer core with a brass extension was permitted to react against the top of the cell. Thus, even though the cell deformed slightly under load, the LVDT measured only the true deformation of the ram and, hence, of the soil sample. The standard calibration procedure of the LVDT (2) was simplified by using a micrometer attachment fitted with an adaptor on the shaft that screwed into the transformer core. Thus, once the null position was found, the range could be set on the galvanometer by raising the core an exact amount (0.1250 in.) with the micrometer. After calibration, the adaptor was unscrewed, the micrometer was removed, and the transformer was ready for use.

VOLUME CHANGE

The technique of measuring volume change was the last to be successfully accomplished. This delay was caused primarily by attempting, during the early development period, to measure volume changes rapidly with respect to time rather than the volume change occurring during the entire test. Accordingly, the first volume change device (Fig. 5) was based on a float principle that recorded the change in cell-fluid level as the test progressed. By relating the incremental increase in fluid with the deformation, and knowing the area of the cap, the volume increase or decrease could be computed. For one test the device was successful in principle but unsuccessful from the test standpoint due to the insufficient range of a 040 LVDT.

An earlier research project by Schaub (4), on volume changes in bituminous mixes during the triaxial test made use of the technique used at Imperial College of Science, University of London (5) (Fig. 6).

In partly saturated soils, a volume change occurs due to densification of the soil and compressibility of the air in the pore space. This change is determined by measuring the volume of fluid enter-

FIG. 6.—Apparatus for Measuring Volume Change Under Pressure.

ing or leaving the cell as the test is conducted. The measurement is made by observing the displacement of a free surface between the water supply to the cell and some other fluid such as mercury. Any change in volume of the soil causes a change in volume of the liquid in the cell which, in turn, is reflected by a change in the level of the mercury in the measuring tube. A differential head of mercury between the mercury reservoir and the measuring tube is avoided by suspending the reservoir on a calibrated spring which changes length directly as the mercury level in the measuring tube changes.

Figure 6 shows the mechanism of the device in detail. With application of pressure to the cell, a decrease in volume of the sample takes place causing the volume of the cell liquid to increase and the level of mercury in the measuring tube to rise. For the soils studied in this research program, a decrease in volume of the soil took place rapidly at the beginning of the test, causing the mercury level to rise further until the volume change stabilized or reached an equilibrium value. In the tests in which failure did not occur and the sample was tested statically to failure, the mercury level was lowered in the measuring tube as volume expansion took place. By reading the metric scale adjacent to the measuring tube, the changes in level of the mercury were read to the nearest millimeter. A change in level of 1 mm corresponded to a calculated volume change of 0.0303 cu in.

The observed displacements in mercury level must be corrected for temperature differentials, expansion of the Lucite cylinder under confining pressure, and for volume changes reflected by movement of the loading ram. So far as temperature changes are concerned, the use of a controlled temperature chamber prevented any volume changes arising from this source. During an increase in cell pressure, the cell expands and causes a volume change that must be deducted from the observed displacement of water. This change was established by conducting calibration tests at various confining pressures. An exact dummy was machined to dimensions of 4.000 in. diameter and 8.000 in. high. This was placed in the cell and the confining pressures applied. It was noted that the change recorded for the various confining pressures occurred almost instantly and did not increase appreciably with time after the first 10 min. Accordingly, the confining pressure was applied for a period of at least 10 min before each test. During loading of the sample, water is displaced by the penetration of the loading ram into the cell causing a volume change that must be added to the observed decrease in volume. This change can be calculated from the area and penetration of the ram for various confining pressures.

Several sources of error are to be avoided in using a device of this type to measure volume change. One is the possibility of entrapment of air in the top of the triaxial cell. Air compressed at high-confining pressures eventually enters into solution, thus causing erroneous volume changes. Another source of error arises from water leakage in the cell and volume-change apparatus; thus all joints must be tight and leakproof. In early tests using this apparatus, some errors resulted in the measured volume changes from both sources. In addition, for tests of long duration, errors may arise from creep in the Lucite cylinder and from leakage through the rubber membrane.

TEST CONDITIONS

The sample to be used was a granular material of small diameter. In addition, the sample would be compacted after laboratory mixing to permit the specimen to be handled. A 4-in. diameter specimen is customary since the same compaction equipment can be used as for the standard compaction test. Furthermore, the measurement of pore pressure using a probe indicated a sample as large as practicable. Normally an *l/d* ratio of 2.00 to 2.5 is used; thus the sample should be at least 8 in. high.

As was mentioned earlier, the accurate measurement of pore pressure can only be accomplished under conditions where the ambient temperature differential is close to zero, so that no pore-pressure changes can be derived from changes in temperature. Thus the equipment was placed in a controlled-temperature environment in which a constant temperature of 0 to 40 C could be obtained. A temperature of 68 F (20 C) was selected for use in testing because it was mainly one of comfort for the personnel, economical for operation, and a standard reference temperature for many materials.

The matter of rate of load application was quite difficult to reconcile. Factors to consider were equipment capability, pore-pressure build-up and dissipation, relation to field conditions, and continuity with previous work. The limitation on equipment was imposed chiefly by the port size of the reducing valve on the main air line and the volume of air necessary to build up the load to its maximum value. From previous experience (3), it was known that some time delay could be expected. To keep this to a minimum, the air cylinder and valves were located as close to each other as possible. Timers were not a problem. The possible range for equipment design purposes was 0.1 to 1.0 sec interval between load applications and 0.5 to 10 secs load duration.

From the standpoint of pore pressures under highways, the rate should be slow enough so that the pore pressure build-up during load would have time to dissipate prior to the next load application, and fast enough to prevent total redistribution of the pore pressures in the sample with load application. If performed too quickly, the response of the equipment might not indicate the true magnitude of the pore pressure change.

Seed, Chan, and Monismith (6) found that for compacted clays the frequency of load application was not related to total deformation. Haynes (7), in his study of the AASHO Test Road base coarse materials, used a frequency of 40 repetitions per min; Larew (8) used 20 cpm. Although some of the early pilot tests were run at 30 cpm, it was found that the equipment performed very well at 40 cpm with 1-sec duration and a $\frac{1}{2}$-sec interval between load applications.

No program of research on soils should attempt to fix limits or assign values to all the variables under consideration; however some intuitive reasoning must be used to set some of the boundary conditions. This was particularly true of the deviator stress used for the specimens. If the stress selected is too high, the specimen will fail under few load applications, and if too low, will result in low strains and pore-pressure values. Accordingly, the deviator stress was fixed on some percentage of the peak-compressive strength of the soil sample as determined from a static triaxial (UU) test. All other test conditions remained the same. Thus the deviator stress was fixed at 30, 40, and 50 per cent of the peak-compressive strength at each confining pressure.

Lastly, the confining pressures selected had to be high enough so that the pore pressure changes would always be positive during the test, but not dangerous to the personnel (7). Although early tests were run as high as 50 psi lateral pressure, this was changed during the test programming to 30, 35, and 40 psi.

SOILS AND MIX DESIGN

For correlation purposes and because of its known properties, Ottawa sand was selected for the aggregate. Ottawa sand has a high degree of sphericity and smoothness in sizes above the No. 100 sieve. Sizes smaller than the No. 100 sieve are produced by crushing; these small sizes are composed of grains having a high degree of angularity and low degree of smoothness. The maximum vibrated dry density of the test sand was 108 lb per cu ft with a corresponding porosity of 0.35. The binder chosen for this study was the minus No. 200 sieve fraction, known locally as the Crosby-B soil. The Crosby-B horizon, a soil very common in Indiana, is derived from glacial drift and described as silt loam. Total clay content averages 35 per cent, of which the majority of the clay mineral is illite and chlorite. Less than 10 per cent is coarser than silt size (0.05mm), and 100 per cent passes the No. 10 sieve. Tests show the liquid limit to average about 34 per cent and the plastic limit 20 per cent.

TABLE I.—SUMMARY OF MIXTURE-GRADATION DATA.

Sieve[a]	Opening, In.	Per Cent Passing, A Mix	Per Cent Passing, B Mix	Per Cent Passing, C Mix
No. 10....	0.0787	100	100	100
No. 16....	0.0469	77.2	80.0	75.8
No. 20....	0.0331	65.0	69.3	62.8
No. 30....	0.0234	54.4	60.0	51.5
No. 40....	0.0165	45.8	52.4	42.3
No. 50....	0.0117	38.6	46.1	34.6
No. 70....	0.0083	32.6	40.8	28.2
No. 100....	0.0059	27.4	36.2	22.7
No. 200....	0.0029	19.0	29.0	14.0

[a] ASTM Specification E 11 – 61.

Of the large number of variables to consider, the binder content was one of the most important. Accordingly, the sand gradation was held constant based on a design gradation computed from Talbot and Richarts' expression $p = 100 \left(\frac{d}{D}\right)^n$ in which $n = 0.5$ as shown by Fuller for maximum density. The computations, using D equal to the No. 10 sieve size resulted in a binder content that was varied as follows: (1) maximum binder required for maximum density, (2) excess binder, and (3) minimum binder. Referring to Table I, the per cent by weight passing each sieve size can be noted ($n = 0.5$).

Thus the per cent binder, or the minus No. 200 sieve fraction, is 19 per cent; this value established the binder content for the A series for which maximum density was the criterion.

From the work of Miller and Sowers (9), the amount of binder required for nearly cohesive behavior was fixed at 10 per cent more than for maximum density or 29 per cent. Miller and Sowers also found that if the volume of voids in the compacted aggregate is assumed to be just filled with compacted binder, the resulting amount was 22 per cent. Similarly they found that the mix exhibited pure cohesive behavior at binder contents above 33 per cent. Applying this relationship to the research led to the decision to use 10 per cent more binder or a total of 29 per cent in an attempt to obtain data for the boundary condition of almost pure cohesive behavior.

Similarly the lower range of 14 per cent binder was arbitrarily fixed as a reasonable lower value for which more nearly cohesionless behavior would be obtained and which at the same time would be high enough to permit relatively uniform saturation of the specimen and handling stability.

Several factors had to be considered in compacting the sample when pore pressures were to be measured that were not applicable to ordinary compaction procedures.

One factor was that of probe placement in a sample containing aggregate compacted to a high density. This had to be accomplished at the time the specimen was compacted, yet the specimen must admit of high or desired density, and 100 per cent (essentially) saturation in order to obtain valid test data.

Chamberlin (10) found that vibration of the mold for 60 to 90 sec on the upper tray of a Gilson mechanical sieving apparatus produced maximum density for open-graded base coarse materials. For open-graded mixes this method is not only satisfactory but nearly mandatory if degradation is to be avoided. When binder is added in any proportion such that the mix is no longer open graded, two possibilities exist. Either the mold and soil can be vibrated together, or dynamic loads can be applied to the soil alone to achieve compaction. The first procedure cannot be effectively used for dense-graded mixes, hence the latter must be used. The greatest difficulty in applying vibration to the soil lies in evaluating the compactive effort or energy imparted to the soil sample.

TABLE II.—MODIFIED AASHO DENSITIES AND OPTIMUM MOISTURE CONTENTS.

Mix	Max Density, lb per cu ft	Optimum Moisture Content, Per Cent	Binder Content, Per Cent
A	136.5	6.35	19.0
B	140.5	6.45	29.0
C	135.5	6.25	14.0

Kneading compaction was given some consideration from the standpoint of the clay-mineral structure formed in a kneading compactor and the similitude with field compaction methods. The clay structure is more oriented in kneading compaction than that produced by impact-compaction tests. Again in the latter, the clay particles are more oriented than those in a static test. Since the binder soil averaged about 35 per cent clay, a total of approximately 6.7 per cent (A Series) of the sample by weight was composed of particles in which the surface forces predominated and the remainder composed of particles in which the gravity forces predominated. Evidently clay structure contributed relatively little to the strength properties of the sample.

It was determined in a comprehensive series of tests that the impact-compaction method conducted in accordance with ASTM Methods D 698 – 58 T[4] produced densities highly consistent in molding water content and in dry density; this method of compaction was used throughout.

Hygroscopic moisture in the binder was determined, and the added water then computed. Densities were computed from oven-dry weight of the sample after test; specific gravity of the soil grains and for the Ottawa sand averaged 2.67 and 2.65, respectively. It will be noted that the compacted moisture contents tended to average about $\frac{1}{2}$ per cent below optimum.

TEST PROCEDURE

Sample Preparation:

The proportion of each fraction making up the design mix was weighed out carefully and thoroughly mixed in the dry state. The amount of water (less hygroscopic moisture in the binder) necessary for compaction was boiled, cooled to room temperature, and mixed into the dry soil.

After mixing was completed, the soil was compacted in the 4-in. diameter by

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[4] Methods of Test for Moisture-Density Relations of Soils, Using 5.5-lb Rammer and 12-in. Drop, *1958 Book of ASTM Standards*, Part 4, p. 1304.

8-in. high split mold. During compaction a brass dummy of the same dimensions as the probe was placed in the soil at mid-depth. The dummy had a threaded-screw hole in the base to facilitate removal when compaction was completed. After carefully leveling the top surface and filling voids from loosened sand grains, the specimen was removed and weighed. As quickly as possible the dummy was removed and the probe which had been de-aired by boiling for 15 min was inserted. The top of the probe minus the cap was covered with a 1-cm square piece of filter paper to restrain sand grains from entering the small orifice and screw holes. Insertion of the probe was easily accomplished since the diameter of the dummy was made 0.005 in. larger than the diameter of the probe. In addition, the round and semi-round grain shape of the Ottawa sand permitted a relatively smooth surface of sliding. The use of a dummy during compaction and removal afterwards was considered to be reasonable on the basis of a remolded soil sample. Subsequent saturation in the saturation cell permitted the probe and soil to reach complete contact throughout.

When the probe had been inserted, the sample was transferred from the base of the split-compaction mold to the base of a split-aluminum saturation mold (Fig. 7). Holes $\frac{1}{8}$ in. in diameter are located at regularly spaced intervals over the periphery of the mold to permit flow of water into the sample. The interior dimensions of the mold are exactly 4 in. in diameter and 8 in. high when assembled.

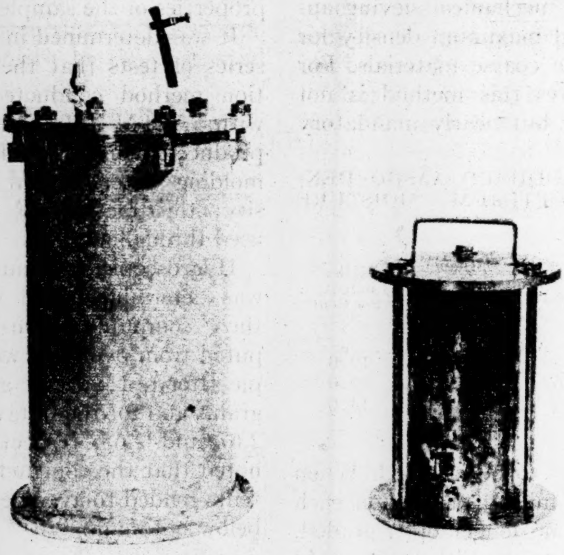


FIG. 7.—Split-Saturation Mold and Saturation Cell.

Previously prepared damp filter paper was wrapped around the sample, smoothed, and the mold clamped tightly around it. The saturation mold containing the sample was then lowered into the saturation tank and covered with de-aired water. A vacuum was then applied for 15 min, after which the hoses were closed securely, and the tank was replaced in the controlled-temperature room for 24 to 48 hr.

Testing:

Each piece of equipment, apparatus, or appurtenance used was kept in the controlled-temperature room at all times. When space requirements demanded, these were removed for only as long as necessary so as to preclude the effect of temperature differentials as a source of error in the results.

Certain other routine procedures were consistently followed such as:

1. The base of the triaxial cell was kept in the controlled-temperature chamber. Prior to use it was submerged in de-aired water and freed of air bubbles in the system by passing de-aired water through the lines repeatedly until no air bubbles remained. All joints were fabricated under water.
2. De-aired water was prepared in advance and stored under vacuum in a 20-liter jar in the controlled-temperature room.
3. A beaker of water containing the two submerged membranes for the next test was kept in the chamber.
4. Chamber temperatures were checked daily by a laboratory thermometer.
5. The Norton-Stone used for the probe was kept in a beaker of water at all times except when in use. Prior to use it was boiled for 15 min to remove any trapped air. Cooling under vacuum in a desiccator jar was attempted and rejected since air was present in the sample.
6. De-aired water was used as the cell fluid in the tests, except where volume change measurements were made.
7. High-vacuum grease was used to

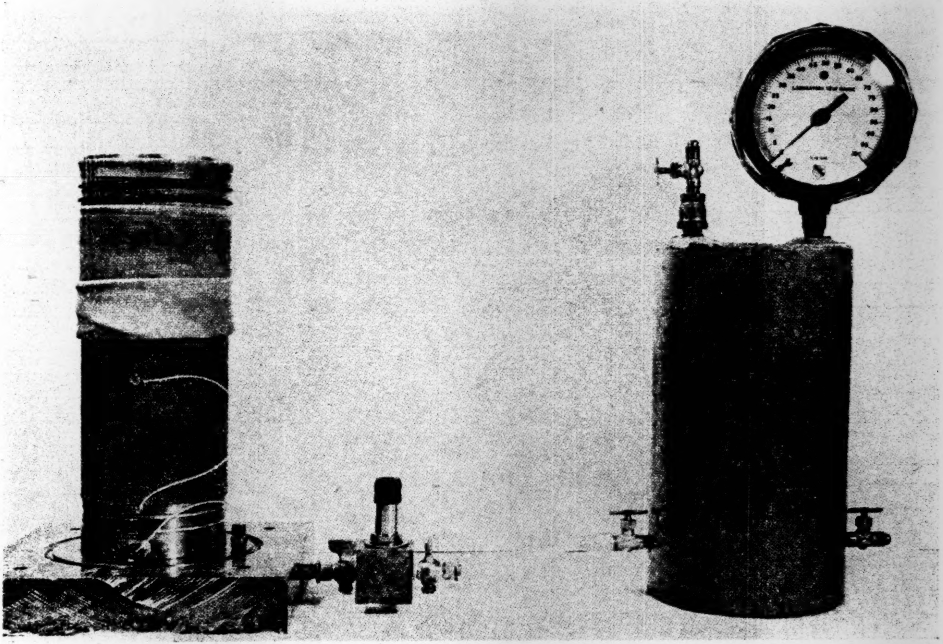


FIG. 8.—Pore-Pressure Sensing System Showing Calibration Tank.

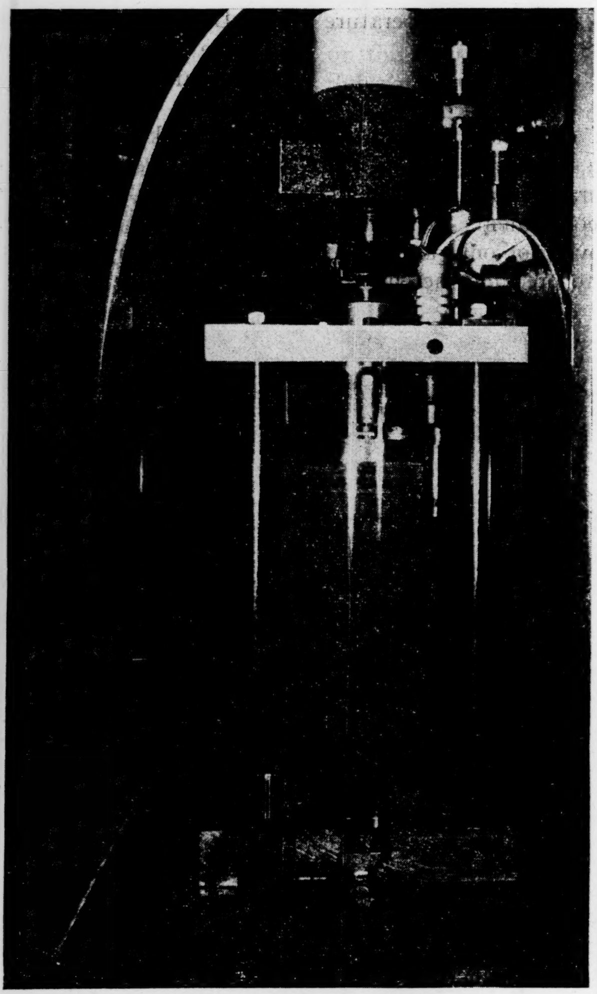


FIG. 9.—Triaxial Cell with Specimen.
Note failure plane developed in test.

lubricate the triaxial cell piston, and all mechanical joints were sealed with suitable compound.

Prior to placing the triaxial cell in the loading apparatus, adjustment of the equipment for the requirements of the test being run was made. The deviator stress or magnitude of applied vertical load was first adjusted by placing an accurate load ring under the load cell and adjusting the dial indicator of the load ring to correspond to the dead load of the piston, ram, and load cell. By adjustment of the main air-line regulator valve, the load was increased or decreased to correspond to the calculated load-ring-dial indicator reading, with the apparatus cycling at test condition. Fine adjustments to the cycling and timing controls were made by observing the load-cell oscillogram. After the deviator stress adjustment, the power

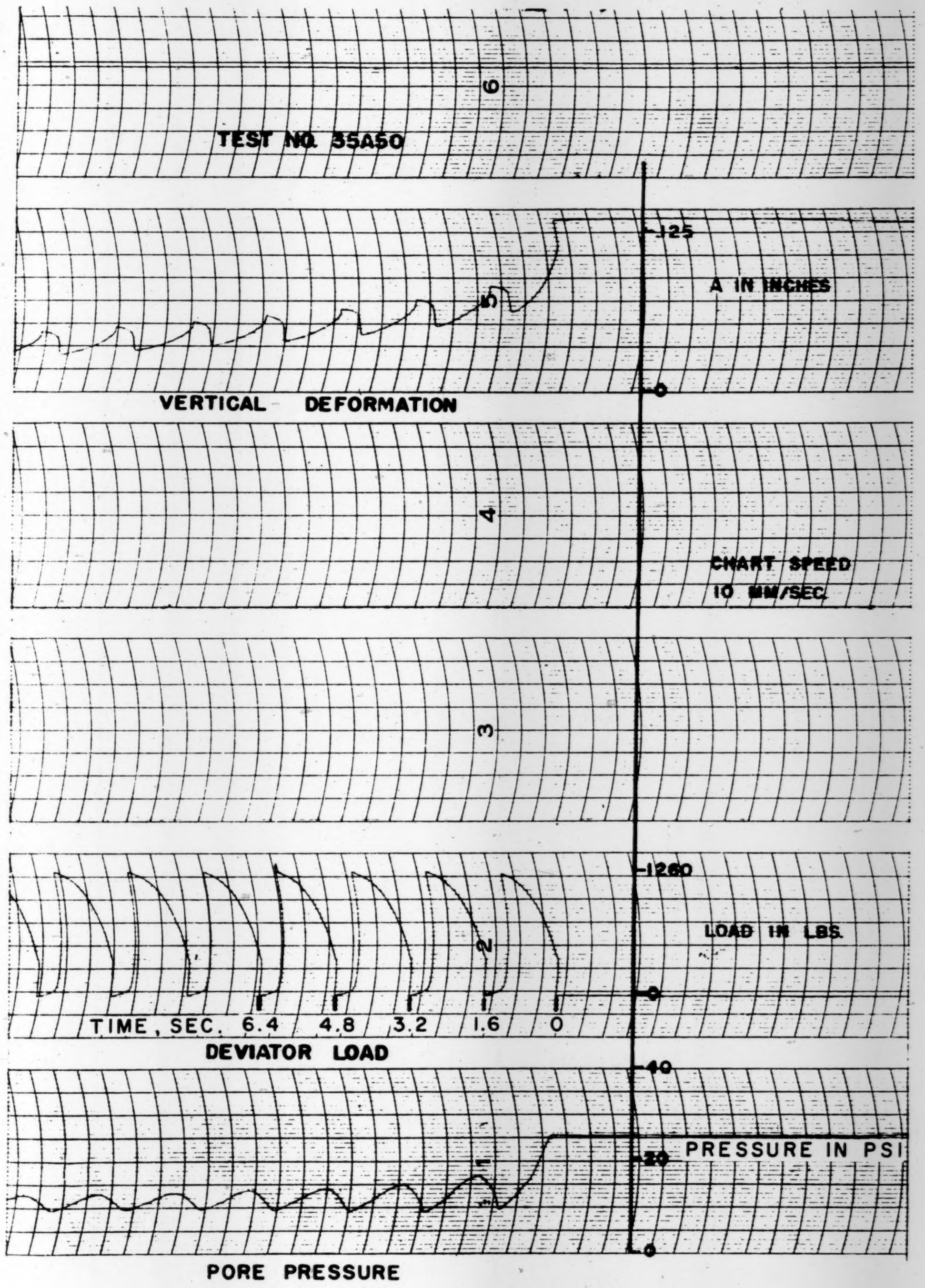
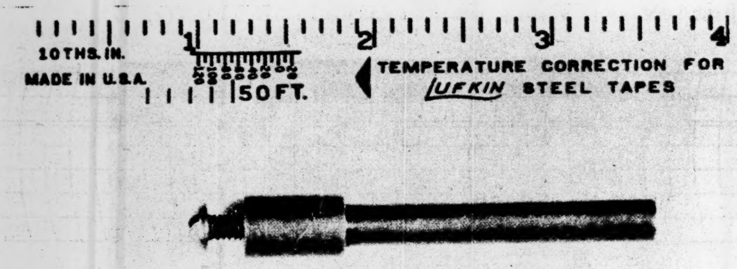


FIG. 10.—Typical Oscillogram Showing Recorded Test Results.


1
2
3
4
10THS. IN.
MADE IN U.S.A.
50 FT.
TEMPERATURE CORRECTION FOR
LUFKIN STEEL TAPES

A. DUMMY PROBE

B. PROBE THROUGH MEMBRANE

C. PROBE UNDER MEMBRANE USED WITH DUMMY

D. PROBE UNDER MEMBRANE COMPACTED IN SPECIMEN

FIG. 11.—Types of Pore-Pressure Probe.

switch was turned off, and the over-ride plunger on the 4-way valve was pushed in and locked. This removed the air from the top of the piston, permitting lifting of the piston and load cell for placement of the triaxial cell. Finally the confining pressure for the test in question was adjusted using the pressure tank calibrator, Fig. 8, and laboratory air gage.

After filling the pore-pressure tubing, the base of the triaxial cell was weighed; the base was again weighed with the sample from the saturation cell. The pore-fluid line was carefully connected to the probe and the probe inserted in the sample. After coating the inside of the triaxial cell with a wetting agent to prevent air bubbles from forming, the specimen, enclosed by two rubber membranes, was placed inside the cell and the cell assembled (Fig. 9). Filling the cell was done with care to avoid entrapment of air. Confining pressure was not applied at this stage unless a compression period was desired; this was the usual case in most tests.

The LDVT for deformation recording was calibrated next according to the procedure described in reference (2). Similarly, the pressure transducer was calibrated using the loading cell method shown in Fig. 6.

Subsequent to recording the zero reading for the volume change device and various dial indicators, the test was started by releasing the over-ride plunger on the 4-way valve. After about 30 cycles, readings were taken at every 10 cycles up to 100 cycles and thereafter at every 100 cycles to 1000 cycles (Fig. 10).

Referring to the pore-fluid-sensing element, Fig. 11 shows a series of four probes. The early tests on A-mixes used probes type B and C. Type B, the very first one used (Fig. 6) was not very successful due to difficulty in preventing leakage around the membrane.

As a result it was decided to place the probe and pore water tube completely under the membranes. Figure 6 shows how this was accomplished. This required some rather refined machine work, but it produced a leakproof joint. The dummy also could be withdrawn after compaction and the probe inserted during the saturation period. This technique did not result in a single failure in making pore-pressure measurements.

In running the tests on the B mixes, it became apparent rather early that considerable volume change was taking place prior to application of the cell-confining pressure. Therefore probe type D was developed which permitted only the pore-fluid pressure in the exact center of volume of the sample to be measured. At the same time, due to the short ceramic portion on the probe, it could be and was compacted into the sample. It worked well for the B mix (29 per cent) binder, but not as well as the type C for the A and C mixes. Further development of the probes is required.

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A STRAIN GAGE PRESSURE CELL FOR RAPID DETERMINATION OF HYDRAULIC CONDUCTIVITY OF SOIL CORES*

By W. C. Bianchi[1] and E. E. Haskell, Jr.[1]

Synopsis

This cell is basically a falling head permeameter where a very small volume of water is displaced through the core as a pressure-stressed metal diaphragm relaxes. The volume displacement of the diaphragm is a linear function of applied head. Head is calibrated against the output of two strain gages applied to the surface of the diaphragm. Recording strain gage output with time on a strip chart recorder or direct reading bridge gives an accurate measurement of the time-head function needed to evaluate the conductivity from the falling head formula.

The determination of hydraulic conductivity on "undisturbed" soil core samples is a time- and space-consuming laboratory procedure. Involved is the measurement of the effluent volume with time under a constant gradient condition (**1**,**5**).[2] Depending on texture and structure, from hours to weeks may be required to displace a measurable volume of water through a core sample of soil. The falling head method (**1**,**5**) can be used to speed up the determination of conductivity in less permeable materials. The lower limit of measurable conductivity below which cores are classed as impermeable, however, depends on how long it is practical to wait to obtain a significant effluent volume.

Principles of Method

This cell is basically a falling head permeameter in which the head-displacement characteristics of a diaphragm are substituted for the vertical uniform-bore tube feeding water to the core. For a circular diaphragm, as in the case of a vertical tube, the change in volume or displacement, v, with change in applied head, h_a, is constant:

$$\frac{dv}{dh_a} = C$$

The displacement constant for a vertical tube is dependent only on its cross-sectional area. With the circular diaphragm, the constant C is dependent on the thickness, diameter, and type of metal used for the diaphragm.

The equation for evaluating the hydraulic conductivity, K, is:

$$K = \frac{LC}{t\pi r^2} \ln \frac{h_0}{h}, \qquad (1)$$

where a saturated core sample of length L and radius r is subjected to a controlled head loss, h_0, until uniform flow is established through the core. The controlled

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* Contribution from the Southwest Branch, Soil and Water Conservation Research Division, Agricultural Research Service, U. S. Department of Agriculture, Fresno, Calif., in cooperation with the California Department of Water Resources.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

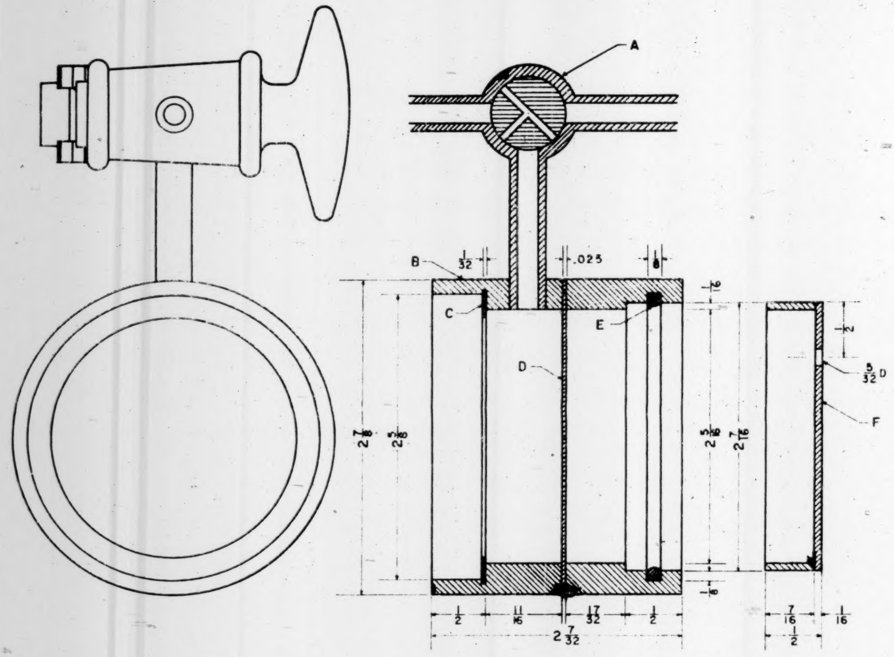
A
B
$\frac{1}{32}$
.025
$\frac{1}{8}$
C
E
D
$\frac{1}{16}$
$2\frac{7}{8}$
$2\frac{5}{8}$
$2\frac{5}{16}$
$2\frac{7}{16}$
$\frac{1}{2}$
$\frac{5}{32}$ D
F
$\frac{1}{8}$
$\frac{1}{2}$
$\frac{11}{16}$
$\frac{17}{32}$
$\frac{1}{2}$
$2\frac{7}{32}$
$\frac{7}{16}$
$\frac{1}{16}$
$\frac{1}{2}$

FIG. 1.—Dimensional Full Section and End View of Cell.

TABLE 1.—PARTS AND MATERIALS NEEDED TO CONSTRUCT A CELL TO MEASURE THE HYDRAULIC CONDUCTIVITY OF 2½-IN. OUTSIDE DIAMETER SOIL CORES.

Parts as Labeled in Fig. 1	Name	Size	Material
A	3-way stopcock	bore of plug 2 mm, side arms 8 mm outside diameter	glass
B	Cell body	2⅞ in. outside diameter by 2$\frac{5}{16}$ in. inside diameter	yellow brass tubing
C	Gasket	$\frac{1}{32}$ in.	red rubber sheet, 75–85 durometer
D	Diaphragm	0.025 in.	brass shimstock
E	O ring	2½ by ⅛ in.	rubber
F	Lid top, side	0.050 in. 2$\frac{7}{16}$ in. outside diameter by 2$\frac{9}{16}$ in. inside diameter	yellow brass sheet yellow brass tubing

head is instantaneously shut off, and the head loss, *h*, is determined after an elapsed time *t*.

TEST APPARATUS

Figure 1 shows a full section and end view of the cell designed to fit soil cores in brass liners 2½ in. in outside diameter. Letters *A* through *F* are identified in Table 1, where individual parts are named and materials are described.

Important details in construction are:

1. The flange on which the gasket rests is inset into the wall of the cell so

that the gasket will not slip from beneath the edge of the liner.

2. The diaphragm is cut from a flat piece of shim stock and care taken not to buckle it at the edges.

3. The diaphragm is soldered between the body parts with 50-50 solder.

4. Two Baldwin-Lima-Hamilton Type FAB-25-12[3] strain gages are applied to the diaphragm surface using EPY 150 cement (2). One gage is placed at the center of the diaphragm to pick up the maximum tangential strain, and the second is located as close to the fixed edge as possible to pick up radial strain there. A half-bridge circuit made up of the two gages allows an increase in sensitivity over a single gage at the center (4). The lead wire, Belden No. 8443, is connected and the gage system waterproofed as suggested by Bean (3). The lead is threaded through the lid, which is then pressed in place. The lead wire is sealed in the hole with a vinyl plastic waterproof cement.

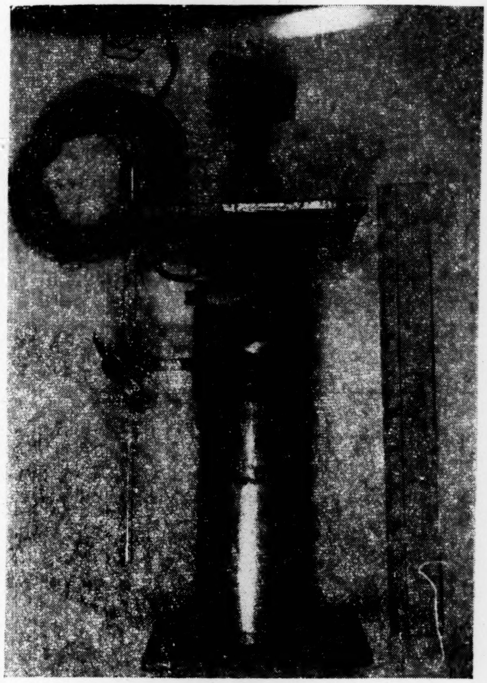

FIG. 2.—Cell and Core Liner in Clamp Assembly.

5. The stopcock *A* is rigidly fixed to the cell with an epoxy cement. The side arms of the stopcock should be in the position shown in Fig. 1.

The complete cell is shown in Fig. 2 in the clamp assembly and fitted to a 6-in. core liner.

The displacement constant *C* for the cell in Eq 1 is determined by rigidly fixing a 0.2-ml pipet, graduated to 0.001 ml, to a closed core cylinder. The cell is clamped to this cylinder under water to eliminate air. A controlled manometer system is used to apply air to the horizontally oriented pipet, and a head-versus-displacement relation is determined from the air-water meniscus in the pipet. Figure 3 shows the displacement relations for two cells: cell 2 made for $2\frac{1}{2}$-in. cores and cell 3 for 2-in. cores. Notice that cell 3 is not linear through the entire pressure range. The constants for these cells, determined by the least squares method, were 5.249×10^{-4} cc per cm H_2O and 2.034×10^{-4} cc per cm H_2O for cell 3 (using the linear portion only).

Simultaneously with the above observations the apparent strain on the diaphragm, as measured by the strain gage system, is calibrated against the applied head. The output of the strain gage network is registered either on a strain-time recorder or a strain indicator bridge. The strain-head function has no theoretical significance and is not linear for these cells; it merely allows one to pick off the head at any instant from a calibrated recorder chart or indicator reading. The upper limit of the calibration, however,

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[3] Trade names and company names are included for the reader's benefit and do not infer any endorsement or preferential treatment of the product listed by the U. S. Department of Agriculture.

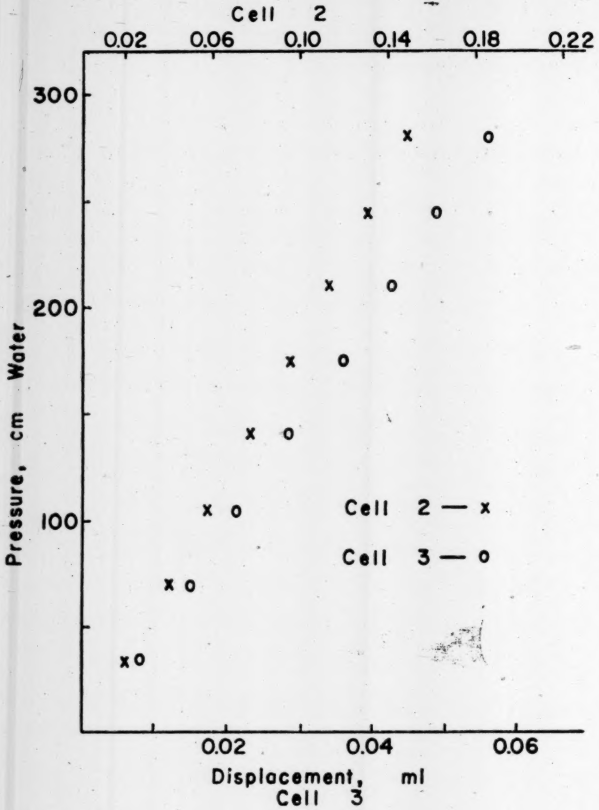

FIG. 3.—Head-Displacement Relations for Cells 2 and 3 (for 2½- and 2-in. Diameter Core Liners, Respectively).

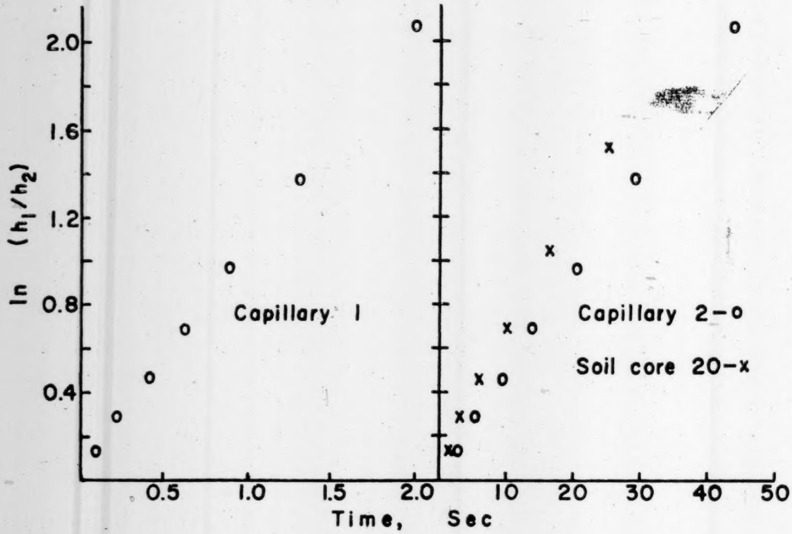

FIG. 4.—Time-Head Loss Relations for Capillary Verification Procedure and for a Soil Core.

TABLE 2.—SAMPLE CALCULATIONS FOR METHOD VERIFICATION.

Cell Observations from Recorder Chart

Cell 2, Capillary Tube 2			Cell 3, Capillary Tube 1		
Head, h, cm H_2O	Time, t, sec	ln h_0/h	Head, h, cm H_2O	Time, t, sec	ln h_0/h
280.8	00.0	0.000	280.8	0.00	0.000
245.7	2.9	0.131	245.7	0.10	0.131
210.6	5.9	0.285	210.6	0.24	0.285
175.5	9.5	0.470	175.5	0.42	0.470
140.4	14.0	0.693	140.4	0.64	0.693
105.3	20.4	0.982	105.3	0.90	0.982
70.2	29.3	1.386	70.2	1.32	1.386
35.1	44.4	2.079	35.1	2.00	2.079
	$S = 0.0468$[a]			$S = 1.032$[a]	

From Equation 1:

$$K \pi r^2 = \frac{LC}{t} \times \ln h_0/h$$

$$= \frac{18.4 \times 5.249 \times 10^{-4} \times 0.0468}{1}$$

$$= 4.52 \times 10^{-4}$$[b]

$$K \pi r^2 = \frac{123 \times 2.034 \times 10^{-4} \times 1.032}{1}$$

$$= 2.58 \times 10^{-2}$$[b]

Discharge Observations

Time, sec	Volume, cc	$\Delta h/L$	Time, sec	Volume, cc	$\Delta h/L$
180	1.226	15.26	120	6.899	2.28
180	1.219	15.26	120	6.932	2.28
180	1.199	15.26	120	6.923	2.28
	Average 1.215			Average 6.918	

$$K \pi r^2 = \frac{V}{t(\Delta h)/(L)}$$

$$= \frac{1.215}{180 \times 15.26}$$

$$= 4.42 \times 10^{-4}$$[b]

$$K \pi r^2 = \frac{6.918}{120 \times 2.28}$$

$$= 2.66 \times 10^{-2}$$[b]

Error

$$\frac{(4.52 - 4.42) \times 10^{-4} \times 100}{4.42 \times 10^{-4}} = 2.26 \text{ per cent}$$

$$\frac{(2.58 - 2.66) \times 10^{-2} \times 100}{2.66 \times 10^{-2}} = 3.10 \text{ per cent}$$

[a] Least squares slope.

[b] Units — $\frac{1}{\text{cm } H_2O \text{ sec}}$

should be below the pressure where displacement becomes nonlinear.

EXPERIMENTAL VERIFICATION OF METHOD

Because of the analogy between Poiseuille's law for the flow through a capillary tube and the Darcy equation for the flow through soil, a length of capillary tube can be treated as an "ideal" soil core. The capillary tube was fixed to the cell system in the same manner as the pipet. The discharge from the horizontal capillary tube (tube 1, approximately ½-mm inside diameter, length 123 cm; tube 2, thermometer tube, length 18.4 cm) was determined for a constant gradient in head. The falling head determination was then made using the cell. The time-head loss data are plotted in Fig. 4. The proportionality constant between the discharge rate and gradient in head was determined by the two methods as per sample calculations shown in Table 2. For capillary tube 1 the difference was 3.10 per cent, and 2.26 per cent for tube 2.

TABLE 3.—HYDRAULIC CONDUCTIVITY OF SOIL CORES MEASURED WITH A STRAIN GAGE PRESSURE CELL.

Core Depth, ft	Elapsed Time, min	Head Loss, h, cm of water	Length of Core, cm	K,[a] cm per min	Remarks
81.5	0.025	245.7	13.3	7.9×10^{-4}	recorder
	0.056	210.6		7.7×10^{-4}	
82.2	0.176	245.7	13.5	1.1×10^{-5}	recorder
	0.413	210.6		1.1×10^{-5}	
97.4	1.83	268.0	12.6	3.8×10^{-6}	bridge
	3.78	252.2		3.9×10^{-6}	
100.3	0.076	245.7	13.9	2.7×10^{-4}	recorder
	0.161	210.6		2.8×10^{-4}	
106.8	13.4	268.0	14.2	4.7×10^{-7}	bridge
	26.0	255.0		7.6×10^{-7}	
118.8	2.03	268.0	13.8	3.8×10^{-6}	bridge
	4.51	252.2		3.6×10^{-6}	

[a] Initial head loss, h_0, was constant at 280.8 cm H_2O. Cell 3 was used which has a cell constant of 2.034×10^{-4} cc per cm H_2O. The 2-in. core liners had a cross-sectional area of 17.9 sq cm.

PROCEDURE FOR SOIL CORES

A tank 15 by 20 by 8 in. is filled with water of approximately the same chemical quality as the soil solution associated with the core samples to be analyzed. The cores, which are at field saturation (or laboratory saturation), are trimmed flush at the bottom, and the upper end is trimmed down about ¼ in. so that the soil will not bear against the gasket of the cell. The cores, cell, and clamp are placed in the solution and allowed to reach a constant temperature.

One arm of the cell's stopcock is connected to the outlet of an aspirator bottle with a length of clear plastic tubing. A zero head reference is established for the cell-core system when the aspirator bottle is filled with solution to a point equivalent to the center line of the core when clamped to the cell in the tank. A regulated air supply is connected to the top of the aspirator bottle. All air is carefully displaced from the tubing and stopcock, and adhering air bubbles are removed from inside the cell and below the gasket.

A core is inserted into the cell and, with the stopcock open to the tank, is clamped firmly in place with its flush lower end bearing on a piece of 50-mesh screen to allow free flow. The zero pressure reference is established on the recorder chart or indicator scale by opening the stopcock to the aspirator bottle which is open to atmospheric pressure. Flow back through the core will not influence this reference unless the soil is very permeable relative to the size of the plastic tubing. Next, the high-pressure reference is determined by applying a measured air pressure to the aspirator bottle. Also, the depth of solution over the center line of the core is kept constant, since this is the exit head from the core. Head loss, h, at any instant, t, across the core is, therefore, the difference between the head as measured indirectly from the strain recorder chart or bridge reading and the exit head.

The time-head function for cores of permeability range, 1×10^{-3} to 1×10^{-5} cm per min, requires a recorder with a chart speed of 2.5 to 25 mm per sec. Start with the maximum head on the core and then turn the stopcock to the "off" position. The times, t, associated with previously calibrated heads are picked from the recorder chart as the pressure and diaphragm displacement are dissipated through the core. In the permeability range, less than 10^{-5} cm per min, it is simpler to use a stopwatch and a direct reading strain indicator bridge to obtain associated time and head readings. The stopwatch is started at the instant the stopcock is closed, and the time for the head to fall through a predetermined head change is determined.

Some typical values of hydraulic conductivity for cores taken from alluvial profiles in Southwestern Fresno County are shown in Table 3. These figures indicate the magnitude of the applied head, the length of time for a determination, and the replication between separate observations during a single run on a core.

In Fig. 4 is a plot of the observed values for the log of the head loss ratio and elapsed time for core 20. The relation closely approaches linearity, but it indicates that conditions within the core deviate slightly from the ideal Darcy relationship.

DISCUSSION

It is possible to prepare and run 20 to 25 saturated cores per day using a single cell. The preparation of unsaturated cores may slow the procedure greatly.

If there is some doubt as to complete saturation in the core, successive determinations can be run until the observed time-head relation becomes constant, or the core may be reversed to see if it remains constant.

The determination can be run on partially filled cores. It must be seen, however, that there is no slippage of the core in the cylinder during the experiment, as this, in effect, adds displacement to the system. Air bubbles have the same effect.

The upper limit of hydraulic conductivity for which the method is directly applicable is in the magnitude of 1×10^{-2} cm per min with a recorder chart speed of 25 mm per sec. It can easily be extended, however, by the use of an auxiliary displacement volume such as a vertical capillary placed in the system. The lower limit of conductivity lies near 1×10^{-8} cm per min; however, it is conceivable that the determination could be extended to 1×10^{-10} cm per min with the use of a thicker diaphragm and instrumentation with strain gages having a gage factor of 100 or more. This latter extension may require further refinements in laboratory temperature control.

The reliability of the hydraulic conductivity as determined through core sampling procedures depends on: (1) how well the core sample represents the soil material from which it was taken, (2) what physical alterations have been produced in the core by the sampling procedure and subsequent handling, and (3) the instrumental errors involved in laboratory procedure.

This procedure gives a minimum of instrumental error even under conditions of very low permeability.

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CORRELATION OF COMPOSITION WITH DURABILITY AND RHEOLOGY OF ASPHALTS*

By Ralph N. Traxler[1]

Synopsis

A modest start has been made in understanding the composition of asphalt. This has resulted in some explanations for the durabilities and viscosities of bitumens manufactured from different sources and by various processes. Well-dispersed asphalts are, in general, most resistant to oxidation and photo-oxidation. Time and temperature are important factors in these reactions. It has been confirmed that oxidation results in the creation of asphaltenes at the expense of resins and in some materials by elimination of saturated hydrocarbons. Chemicals that are free radical inhibitors have been found to reduce the susceptibility of certain asphalts to hardening by oxidation. Waxes and other paraffin hydrocarbons cause a reduction in high-temperature (140 to 275 F) viscosities. All of these effects are shown to be responsible for the qualities of paving cements. Also, they should aid in evaluating bitumens manufactured for other uses.

For many years interest in the composition of asphalt was dormant because most investigators believed the chemistry of asphalt to be so complex that little progress could be expected even with the application of great effort and expense. However, about 20 years ago there was an upsurge of interest based on the use of solvent fractionation. Additional work was stimulated later by the availability of other techniques for fractionation. These methods were, in most cases, borrowed from other fields of technology and included chromatography, thermal diffusion, urea adduction, molecular distillation, and certain chemical reactions.

Solvent fractionation and chromatography were first used by Marcusson (**1**)[2] in an effort to distinguish between native and petroleum asphalts. Solvent fractionation was used by a number of later investigators (**2–5**) in an effort to characterize an asphalt and to yield data on composition that would correlate with physical properties. O'Donnell (**6**) used these and other techniques in an intensive investigation of one asphalt. His work has been extended by others (**7**) to include relationships between composition and properties. During the same period procedures were improved for obtaining elemental analyses (**8**) and data were obtained on the amounts of functional groups present in asphalts.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

Infrared spectroscopy has been helpful when applied to fractions separated by selective solvents, chromatography, and thermal diffusion (9). The quantity of free radicals present in an asphalt undoubtedly has a definite bearing on its susceptibility to change by the various environments encountered in service. This aspect of asphalt chemistry has been studied recently (10, 11). There is little doubt that much progress will be made by use of the new techniques and procedures now available to the chemist, although progress may be slow because of the limited number of investigators working in this field.

TABLE I.—CORRELATION OF DISPERSION COEFFICIENTS WITH DURABILITIES.

Asphalt	Coefficient of Dispersion[a] $X = \frac{R+C}{A+S}$	Susceptibility to Oxidation, Relative Viscosity[b]	Susceptibility to Photooxidation, Relative Viscosity[c]
85 TO 100 PENETRATION ASPHALTS			
No. 6	1.22	2.1	3.4
No. 4	1.03	2.5	10.1
No. 5	0.96	2.8	4.5
No. 10	0.94	3.3	10.1
No. 11	0.67	3.3	8.3
No. 9	0.49	3.5	7.5
No. 3	0.55	4.3	8.5
No. 8	3.61	4.4	8.4
No. 2	0.49	4.5	12.4
No. 1	0.50	6.0	13.9
No. 7	0.59	6.2	13.7
120 TO 150 PENETRATION ASPHALTS			
No. 6A	1.78	2.5	3.9
No. 11A	0.79	2.6	5.3
No. 3A	0.65	4.0	6.0
No. 1A	0.61	4.2	11.2

[a] R = resins, C = cyclics, A = asphaltenes, S = saturates.

[b] 15-μ films oxidized in dark air oven for 2 hr at 225 F.

[c] 15-μ films oxidized under radiation from 175-w sunlamp for 2 hr at 190 F.

The purpose of this paper is to report efforts that have been made to correlate data on composition now available with new information concerning the flow properties of asphalts and their susceptibility to hardening by the environments encountered under service conditions. Much of the more precise chemical information available is not sufficiently complete to use in such correlations.

Extensive durability and rheological data have been obtained at The Texas Transportation Inst. on a considerable number of commercial paving asphalts (12, 13). Several of the available methods of analyses (4, 5, 14), or modifications of them, have been applied to representative asphalts in an effort to establish correlations which might help in predicting the durability and flow properties of these asphalts. The same principles should be applicable to asphalts used for other purposes.

CORRELATION OF DURABILITY WITH COMPOSITION

The investigations mentioned above were conducted to determine the susceptibilities of asphalts from various sources, made by different processes, to hardening by oxidation, photooxidation, volatilization, and so on. First, the viscosity of the asphalt was measured in the microfilm (sliding plate) viscometer[3] at 77 or 95 F. Then, 15-μ films of the asphalt were placed on glass plates and exposed to a selected environment. The hardened film was scraped from the plate and its viscosity measured at the temperature used with the original asphalt. The viscosity of each hardened film was divided by the viscosity of the original asphalt to yield a quotient called the relative viscosity, which quantitatively evaluates the hardening under the environmental conditions used.

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[3] This type of apparatus is sold by Hallikainen Instruments, Berkeley, Calif.

The procedure used is similar in principle to that proposed by Subcommittee B-19 of ASTM Committee D-4 on Road and Paving Materials. In this test a 5-μ film is exposed to air in the dark for 2 hr at 225 F, and the increase in viscosity is determined. In our investigations 15-μ films were used because that is about the average thickness of the binder film in a bituminous pavement. In the photooxidation test it was convenient to conduct the experiments at 190 F.

Whether treatment of the asphaltic film is exposure to air in the dark (oxidation), exposure to air in the presence of actinic light (photooxidation), or exposure to an inert atmosphere such as nitrogen (volatilization), the effects of time and temperature are very important.

Data obtained by a modification of the component-analysis method (**5**) were used to calculate a coefficient of dispersion, *X*. Four fractions are obtained: (1) asphaltenes (*A*); (2) resins (*R*); (3) saturates (*S*); and (4) cyclics (*C*). This method of solvent fractionation involves precipitation of the asphaltenes and resins by digesting the asphalt for 30 min with 20 parts of boiling 1-butanol and then maintaining the mixture at 122 F for 16 to 18 hr. The hot solution is filtered, and the insoluble, or asphaltic, material is washed repeatedly with hot 1-butanol. A standard determination of asphaltenes, *A*, is made on another sample of asphalt and the amount of asphaltenes is subtracted from the asphaltics to give the amount of resins, *R*, present in the sample. The solution of oil in 1-butanol is distilled to remove the solvent. The recovered oil is refluxed for 15 min with 35 parts of acetone, allowed to cool in the room, and then chilled for 16 to 18 hr at −10 F. The solution is decanted from the precipitated saturates, *S*, which are predominantly paraffinic hydrocarbons. Finally, the solution is stripped of acetone leaving the cyclics, C, which are chiefly aromatic-type materials.

TABLE II.—CORRELATIONS OF RELATIVE VISCOSITIES WITH DISPERSION COEFFICIENT CALCULATED FROM 1-BUTANOL-ACETONE AND ISOPROPANOL-ACETONE DATA. 120 TO 150 PENETRATION ASPHALTS.

Asphalt	Fractionation Procedure	Composition, per cent				Coefficient of Dispersion	Relative Viscosity[a]
		Asphaltenes	Resins	Saturates	Cyclics	$X = \frac{R+C}{A+S}$	
No. 1A......	1-butanol	11.0	21.0	51.0	17.0	0.61	4.2
	isopropanol	11.0	30.2	36.2	22.6	1.15	
No. 3A......	1-butanol	17.5	25.5	43.0	14.0	0.65	4.0
	isopropanol	17.5	41.0	25.0	16.5	1.35	
No. 6A......	1-butanol	1.0	55.0	35.0	9.0	1.78	2.5
	isopropanol	1.0	90.0	6.0	3.0	13.0	

[a] Relative viscosity of 15-μ film oxidized in the absence of actinic light for 2 hr at 225 F. Viscosities measured at 77 F and calculated at a rate of shear of 5×10^{-2} sec^{-1}.

Resins and cyclics are dispersing agents for the asphaltenes, whereas the saturates are coagulating agents (**12**) and thus tend to counteract the effect of the resins and cyclics. The degree of dispersion, *X*, within the asphalt may be expressed by the ratio $(R + C)/(A + S)$. A well-dispersed or sol-type asphalt contains an excess of resins and cyclics (large value for *X*). A poorly dispersed or gel-type asphalt contains an excess of asphaltenes and saturates (small value for X).

Table I shows fair correlation between the susceptibility of an asphalt to hardening by oxidation and the degree of dispersion, X, for several asphalts of different sources, consistencies, and methods of production. One extreme exception is asphalt 8. This is a solvent-refined material of unusual composition. More precise procedures than the component analysis method may result in the establishment of better correlations between composition and resistance to oxidation.

TABLE III.—CORRELATION OF ROSTLER AND STERNBERG DATA WITH SUSCEPTIBILITY TO OXIDATION.

Asphalt	Relative Viscosity[a]	Coefficient X[b]	Rostler Coefficient[c]	Modified Rostler Coefficient[d]
85 TO 100 PENETRATION ASPHALTS				
No. 1......	6.0	0.50	1.19	1.50
No. 3......	4.3	0.55	0.90	1.45
No. 11......	3.3	0.67	0.75	1.76
No. 6......	2.1	1.22	1.33	5.65
120 TO 150 PENETRATION ASPHALTS				
No. 1A.....	4.2	0.61	1.05	2.68
No. 3A.....	4.0	0.65	1.03	2.46
No. 11A.....	2.6	0.79	0.97	2.68
No. 6A.....	2.5	1.78	1.49	8.70

[a] Relative viscosity of 15-μ films oxidized in dark oven for 2 hr at 225 F.

[b] $\dfrac{\text{resins} + \text{cyclics}}{\text{asphaltenes} + \text{saturates}}$

[c] $\dfrac{\text{nitrogen bases} + \text{1-acidaffins}}{\text{paraffins} + \text{2-acidaffins}}$

[d] $\dfrac{\text{nitrogen bases} + \text{1-acidaffins} + \text{2-acidaffins}}{\text{asphaltenes} + \text{paraffins}}$

The final column of Table I shows that a somewhat less satisfactory correlation exists between dispersion coefficient X and photooxidation as evaluated by relative viscosity. This is not surprising since photooxidation is probably more involved with the chemical composition of the asphalt than is oxidation in the absence of actinic light. Asphalts 4 and 10 are well-dispersed materials (high values for X) but are quite susceptible to hardening by photooxidation; that is, each has a relative viscosity of 10. Also, asphalt 8, which is very well dispersed ($X = 3.6$), is not particularly resistant to photooxidation (relative viscosity = 8.4).

These exceptions do not invalidate the concept that a relationship exists between degree of dispersion and resistance to hardening. They do, however, indicate that much additional work needs to be done in the field of asphalt chemistry.

A number of the asphalts listed in Table I were fractionated by the Pitchford and Axe method (4). Three fractions are obtained: (1) asphaltenes (A); (2) resins (R); and (3) oils (O). Asphaltenes are precipitated by n-pentane and filtered from solution. Pentane is distilled from the solution and the recovered petrolenes refluxed for 2 hr with about 50 parts of isopropanol. This solution is filtered, and the precipitate, called resins, is washed with hot isopropanol. The isopropanol solution is distilled to recover the oils. No combination of these fractions correlated with the susceptibility of an asphalt to oxidation as measured by relative viscosity. However, by fractionating the oils with cold acetone, as is done in the component-analysis procedure, a useful dispersion coefficient could be calculated. Although for each asphalt the value of X was higher than the values shown in Table I, good dispersion was again associated with resistance to oxidation. Data on three soft asphalts are shown in Table II.

Several of the asphalts were analyzed by the Rostler-Sternberg sulfuric acid method (15) and the coefficient proposed by Rostler and White (14) calculated for each asphalt. Briefly this method comprises removal of asphaltenes by *n*-pentane followed by treatment of the petrolenes successively with 85 per cent, 98 per cent, and fuming sulfuric acids. Five basic fractions are obtained: (1) asphaltenes (A); (2) nitrogen bases (N); (3) 1st acidafins (A_1); (4) 2nd acidafins (A_2); and (5) paraffins (P).

The Rostler and White coefficient, $(N + A_1)/(P + A_2)$, did not correlate with the relative viscosities of the 15-μ films oxidized for 2 hr at 225 F. Additional calculations were made using combinations of the five fractions, but no better correlations were obtained. It was concluded from the data obtained on eight different asphalts (Table III) that the sulfuric acid method does not help in predicting the susceptibility of an asphalt to hardening by oxidation. Further refinements in the procedure may overcome some of the difficulties encountered.

CHANGE IN COMPOSITION CAUSED BY OXIDATION

There have been numerous studies (9) concerning the oxidation of asphalt. Thurston and Knowles (16) investigated the effect of oxygen in a closed system on asphalts and asphaltic components such as asphaltenes, resins, and oils. They found that the asphaltenes are readily oxidized when they are separated from the protective action of the resins (and presumably some of the oils). The belief has become established that oxidation of asphalt results mainly in the production of asphaltenes at the expense of the resins present. To obtain some evidence concerning this, the following experiments were conducted.

Six representative 85 to 100 penetration-grade paving asphalts were oxidized in the form of ⅛-in. (3175-μ) films for 15 hr at 325 F. This is essentially the Bureau of Public Roads Thin-Film Test except the time of oxidation was increased from 5 to 15 hr. Each oxidized sample was thoroughly mixed while hot, and a portion was analyzed by the modified component-analysis method (5). As would be expected from the past work of numerous investigators, the oxidized samples showed a marked increase in asphaltenes over the original (Table IV). Likewise, each showed a decrease in resin content, which further supports the idea that the resins are the chief source of asphaltenes created by oxidation. No clear-cut deductions can be drawn concerning the effect of oxidation on the oils, although in the case of asphalts 2 and 3 it appears that heavier materials are created at the expense of saturates. Here is another example of the need for more precise chemical information. The behavior of the saturates in asphalts 2 and 3 might be explained if more were known about their chemical composition.

TABLE IV.—CHANGE IN COMPONENTS RESULTING FROM OXIDATION OF ⅛-IN. FILMS OF 85 TO 100 PENETRATION ASPHALTS FOR 15 HR AT 325 F.

Asphalt	Asphaltenes	Resins	Saturates	Cyclics
No. 1	+9.2	−12.2	+1.0	+2.0
No. 2	+6.0	−3.0	−6.0	+3.0
No. 3	+5.1	−5.1	−2.5	+2.5
No. 4	+12.2	−15.9	+3.3	+0.4
No. 5	+9.3	−10.3	+1.0	0.0
No. 6	+9.0	−9.0	0.0	0.0

FREE-RADICAL INHIBITORS AS ADDITIVES TO RETARD OXIDATION OF ASPHALTS

Additives have been used in asphalts for years to improve their adhesion to damp surfaces and to retard stripping from solid surfaces by water action. Also, there is need in the asphalt industry for materials that can inhibit the hardening of asphalt by oxidation. Little work has been done on this subject, and the materials tried in the past have not been effective enough to be economically useful. In recent months the thin film techniques discussed above have been used at The Texas Transportation Inst. in the evaluation of a number of additives supplied by two manufacturers.

TABLE V.—EFFECT OF ADDITIVE B-8 ON ASPHALT 1, 85 TO 100 PENETRATION GRADE.

Specimen	Viscosity, poises at 77 F	15-μ Films Heated for 1 hr at 275 F			
		In Air		In Nitrogen	
		Viscosity, poises at 77 F	Relative Viscosity	Viscosity, poises at 77 F	Relative Viscosity
Asphalt No. 1	1.1×10^6	12.5×10^6	11.4	3.0×10^6	2.7
No. 1 + 1 per cent B-8......	1.1×10^6	6.6×10^6	6.0	3.1×10^6	2.8

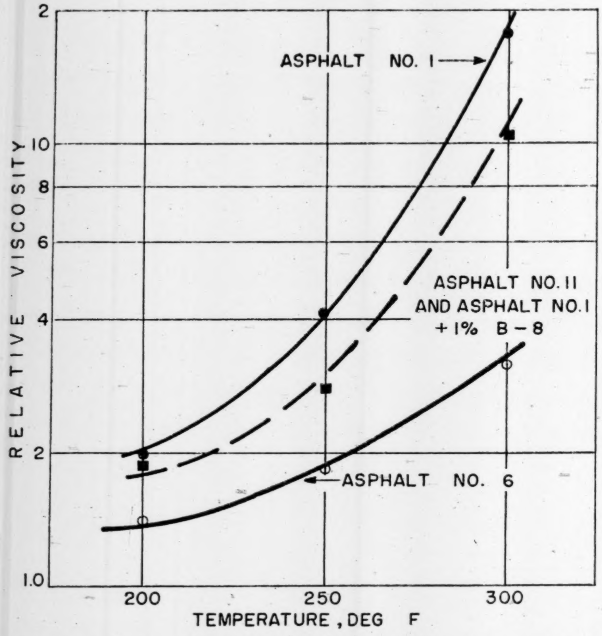

FIG. 1.—Oven Oxidation of 15-μ Films; 1-hr Effect of Additive B-8 on Relative Viscosities. Rate of shear, 5×10^{-2} sec^{-1}.

A weighed amount (up to 3 per cent) of each additive was thoroughly blended with two different 85 to 100 penetration grade paving asphalts. Asphalt 1 is highly susceptible to oxidation and asphalt 11 is moderately resistant. Asphalt 6, which is very resistant to hardening by oxidation, was used for comparison with the blends. Care was taken that the mixtures were not overheated during compounding. The sliding plate (microfilm) viscometer was used to determine the viscosities of the blends at 77 or 95 F. Viscosities were calculated at a rate of shear of 5×10^{-2} sec^{-1}.

Next, a 15-μ film of each blend was placed on a 4 by 4-cm glass plate and exposed to air in the presence and absence of sunlight and to nitrogen in the absence of actinic light at various temperatures for different times. The hardened asphalt was scraped from the glass surface and placed between the 2 by 3-cm plates used in the sliding plate viscometer. The viscosities of the hardened asphalt-additive blends were determined at the same temperature as that used for evaluating the original untreated blends. The viscosity of oxidized (hardened) blend was divided by that of the untreated blend to obtain the relative viscosity of the hardened sample. This value was compared with the relative viscosity of the original asphalt exposed to the same environment to determine the effectiveness of the additive. If the relative viscosity of the treated blend was significantly less than that of the treated original asphalt, the additive was considered to be worth further consideration.

Of the 16 additives investigated, one, coded as B-8, was superior to the others in its ability to inhibit oxidation in an asphalt which is highly susceptible to hardening by oxidation. Thus data obtained on blends containing B-8 will be used to illustrate the kind of results obtained. The manufacturer classifies this additive as a free-radical inhibitor. To date, we have had no opportunity to evaluate the free-radical concentrations of the asphalts and blends, but it seems likely that a part of the oxidation occurring in an asphalt is related to the amount of free radicals present.

Table V shows the effect of 1 per cent of additive B-8 in asphalt 1 when 15-μ

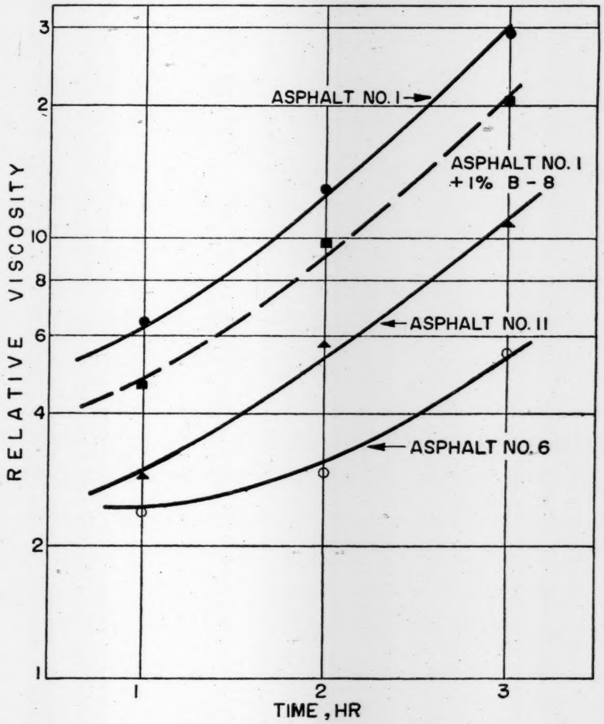

FIG. 2.—Photooxidation of 15-μ Films at 190 F; Effect of 1 Per Cent Additive B-8 on Relative Viscosities.

Rate of shear 5×10^{-2} sec^{-1}.

films were heated (1) for 1 hr at 275 F in an air oven in the absence of actinic light, and (2) for 1 hr at 275 F in an atmosphere of nitrogen and in the absence of light. A considerable reduction in susceptibility to hardening by oxidation, as measured by relative viscosity, was effected in asphalt 1 by the small amount of additive B-8 used. Tests made in an atmosphere of nitrogen showed that the additive did not materially affect hardening due to volatilization.

TABLE VI.—EFFECT OF PARAFFIN WAX ON VISCOSITY OF A PAVING ASPHALT.

Asphalt	Penetration at 77/100/5	Viscosity, stokes at	
		140 F	175 F
No. 6	85	1380	155
No. 6 + 5 per cent wax	69[a]	340	55
No. 6 + 10 per cent wax	46[a]	90	20

[a] These blends possessed so much gel structure at 77 F that the cavity created in a can of the asphalt by cutting out a sample would not be filled, during the elapse of several weeks, by flow of the remaining asphalt.

Figure 1 is a semilogarithmic plot illustrating the hardening (increase in relative viscosity) of asphalts 1, 11, and 6, and of a blend of asphalt 1 with 1 per cent of additive B-8 oxidized for 1 hr at temperatures from 200 to 300 F. The additive reduced the susceptibility of asphalt 1 to that of asphalt 11 without additive. Considerably more additive would be required to make asphalt 1 as resistant to oxidation as asphalt 6. The viscosity data in Fig. 1 were obtained at 95 F.

Photooxidation tests were made on the three asphalts and the blend mentioned above. Results are shown in Fig. 2, also a semilogarithmic plot. These curves indicate that the additive is not as effective in inhibiting hardening caused by photooxidation as it is in reducing oxidation in the absence of actinic light. In the experiments illustrated in Fig. 2, tests were made on 15-μ films for 1, 2, and 3 hr at 190 F. Viscosities were measured at 95 F. A 175-w sunlamp placed 11 in. from the asphalt film was used as the source of radiation.

TABLE VII.—CORRELATION OF COMPOSITION WITH VISCOSITY.

Asphalt	Per Cent Saturates[a]	Viscosity, stokes at		
		140 F	175 F	275 F
85 TO 100 PENETRATION ASPHALTS				
No. 8	23	2580	260	7.0
No. 3	40	2600	250	5.4
No. 10	40	2630	205	4.8
No. 4	40	1810	195	4.3
No. 6	44	1380	155	3.5
No. 11	49	1630	160	3.4
No. 5	43	1030	110	3.3
No. 9	50	1450	150	3.1
No. 2	48	1420	145	3.1
No. 1	49	1250	125	3.0
No. 7	48	1010	105	2.6
120 TO 150 PENETRATION ASPHALTS				
No. 6A	35	1040	135	4.2
No. 3A	43	1150	150	3.8
No. 11A	45	1120	120	3.1
No. 1A	51	650	75	2.3
No. 7A	47	560	65	1.9

[a] 1-butanol soluble-acetone insoluble fraction of the asphalt.

The general conclusion to be drawn from this series of experiments is that chemicals are available that are capable of inhibiting to some extent the oxidation of asphalts. Further work along this line may result in economic benefit to the bituminous industry.

EFFECT OF PARAFFIN HYDROCARBONS ON THE VISCOSITY OF ASPHALT

When the First World Petroleum Congress was held 30 years ago considerable interest was shown concerning the effect of paraffin wax on the properties of asphalt. Rheim (17) found that the presence of wax lowered the viscosity and adhesiveness of asphalts. He concluded that 5 per cent was the maximum amount of wax that could be tolerated in an asphalt. Recent work has demonstrated the profound effect that the addition of 5 to 10 per cent of paraffin wax has on the high-temperature viscosity of an asphalt.

Table VI gives data obtained in a Cannon-Manning vacuum capillary viscometer. The asphalt used was an 85 to 100 penetration grade material. It will be noted that the addition of 5 and 10 per cent by weight of "grocery store" wax caused a tremendous decrease in viscosity at 140 and 175 F. At these temperatures, which are critical in the rolling and compaction of a bituminous paving mixture, the presence of 5 per cent wax resulted in a blend only about one fourth as viscous as the original product. The viscosities at 140 F of the blends (Table VI) are much below the range tentatively established by the Asphalt Inst. for 85 to 100 penetration grade asphalts.

To determine whether the amount of paraffin (saturated) compounds in an asphalt affects its viscosity at high temperature, component analyses were made on 16 asphalts from various sources and producers. Table VII compares the per cent of saturates (predominantly paraffin compounds) in each asphalt with its kinematic viscosity at 140, 175, and 275 F. Asphalts 5 and 7A are out of line. These discrepancies may be removed when we have developed a more accurate method for determining the saturated hydrocarbons in an asphalt. It is logical to assume that a high paraffin content would result in greater susceptibility to temperature change and thus a lower viscosity at the high temperatures as shown in Table VII.

SUMMARY

From the work described in this paper, it may be concluded that fairly satisfactory correlations exist between hardening of thin films by oxidation and the composition of asphalt determined by modifications of the Traxler-Schweyer and Pitchford-Axe procedures. Oxidation of asphalt in air at 325 F results in formation of asphaltenes at the expense of resins and in some cases reduction in the amount of saturated compounds. Chemicals classified as free-radical inhibitors markedly reduce the susceptibility of certain asphalts to hardening by oxidation. Saturated hydrocarbons (waxes and oils) in asphalts result in a reduction in high-temperature viscosity. All of these effects have an important bearing on the quality of asphalts for various uses.

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(13) R. N. Traxler, "Durability of Asphalts," presented at Annual Meeting Assn. Asphalt Paving Technology, San Francisco, Calif., Feb. 18–20, 1963.

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DISCUSSION

MR. F. S. ROSTLER[1] (*by letter*).—Mr. Traxler's paper contains a number of statements which contradict well-established facts and presents conclusions not substantiated by the author's data, most of which are not even related to the phenomena discussed. The data reported also make the accuracy and reliability of the experimentation highly questionable. The design and outline of the test program is unrealistic and deals with material never considered for use in highway construction.

The author stated that:

1. "The investigations . . . were conducted to determine the susceptibility of asphalts . . . to hardening by oxidation, photooxidation, volatilization, and so on." This statement is misleading since the author did not test the phenomena enumerated. The definitions given ("Oxidation is exposure to air in the dark; photooxidation, exposure to air in the presence of actinic light; volatilization, exposure to an inert atmosphere") are evidence that the author uses chemical nomenclature very loosely and phrases his conclusions in terms far outside the observations made. The author does not realize that all he observed was hardening under the conditions described, not the phenomena labeled "oxidation," "photooxidation," and "volatilization."

2. "A modification of the component-analysis method was used to calculate a coefficient of dispersion." The author did not measure any dispersion and therefore could not show any correlation between dispersion and the data obtained in the analysis; he merely assumed the existence of a correlation. It is noteworthy that Traxler refers to his method as *"the*

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[1] Vice President of Product Development and Director of Research, Golden Bear Oil Co., Bakersfield, Calif.

component analysis." This status cannot be accorded to the Traxler-Schweyer method which has been shown[2] to yield data not related to asphalt composition and to be at variance with all other methods of analysis based on scientific principles of fractionation. The modified Traxler-Schweyer method is neither a fractionation nor a suitable basis for calculation of composition. Apparently in response to the above quoted paper, in which was shown that all so-called fractions of the Traxler-Schweyer method contain various amounts of all components including asphaltenes, the modified method was devised which consists of determining the asphaltenes on a separate sample and subtracting the amount from the first fraction. This "improvement" results with some asphalts in a negative value for per cent resins.

3. "The precipitated saturates, *S*, are predominantly paraffinic hydrocarbons." Work by others and the data presented by Traxler in Table VII of the paper for the specimens tested prove that this is not the case. Petroleum residues containing 40 to 50 per cent saturates are not asphalts. If produced, they would be incompatible mixtures of cheesy consistency, with no resemblance to highway asphalts.

4. "The cyclics, *C*, are chiefly aromatic type materials." This again is an assumption without justification repeatedly proven false. Traxler's fraction *C* is a mixture of all known asphalt components, including asphaltenes.

5. "A relationship exists between degree of dispersion and resistance to hardening." Nowhere has this relationship been shown by Traxler. He merely quotes a statement made by Mertens[3] without any credit to the author and his work.

6. "It was concluded from the data obtained on eight different asphalt specimens that the sulfuric acid method does not help in predicting the susceptibility of an asphalt to hardening by oxidation." Extensive investigations based on several hundred asphalt specimens have shown that a very good correlation exists between hardening and composition determined by this method.[4] The limited number of asphalt specimens tested (if they were asphalts) does not permit drawing this conclusion in the face of the overwhelming evidence to the contrary. Since Traxler did not report the analytical data obtained, his conclusions might also be suspect of being based on erroneous results.

7. "Heavier materials are created at the expense of saturates." This is a completely meaningless statement without proof or any chemical foundation. Traxler's observation proves that his fraction *S* does not constitute paraffinic hydrocarbons.

8. "Free radical inhibitors markedly reduce the susceptibility of certain asphalts to hardening by oxidation" and "oxidation occurring in an asphalt is related to the amount of free radicals present." Traxler neither showed the existence of free radicals in the asphalt, nor supplied any evidence that the material used was a free radical inhibitor, except that it was so named.

9. "It is *logical* to assume that a high paraffin content would result in greater susceptibility to temperature change" of viscosity. This assumption is, on the contrary, *illogical* and contradictory to all experience in petroleum chemistry

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[2] Fritz S. Rostler and Richard M. White, "Influence of Chemical Composition of Asphalts on Performance, Particularly Durability," Symposium on Road and Paving Materials—1959, *ASTM STP No. 277*, 1959, p. 64.

[3] E. W. Mertens, "Predicting Weatherability of Coating Grade Asphalts from Asphaltene Characteristics," *ASTM Bulletin*, No. 250, Dec., 1960, p. 40.

[4] *Proceedings*, Am. Assn. Paving Technologists, Vol. 31, 1962, p. 35.

where a flat temperature-viscosity curve has long been found to be dependent on a high amount of saturated hydrocarbons. The proof provided by Traxler by adding "grocery store" wax to one asphalt is not even related to the phenomenon discussed nor to the saturated fractions determined by the Traxler-Schweyer method. Footnote *a* in Table VI of the paper reveals Traxler's misconceptions regarding influence of asphalt composition on properties. The "gel structure" observed by Traxler is obviously paraffin crystals in a nonhomogeneous mass.

The above points are only the most glaring fallacies. Since papers presented in ASTM meetings and printed in ASTM publications have the objective of disseminating and not of dissipating scientific information, it is suggested: (1) that the author confine his conclusion to what he found; (2) that he recheck his data; (3) that he publish complete information on the specimens tested—for example, all the analytical data, not only parameters calculated from the data, and (4) that he make available to others samples of the asphalts tested so that the accuracy of observations and measurements can be checked.

Specifically, I should like to receive specimens of the materials tested by the author and will then gladly report my findings. In case the author should not have any specimens left, I suggest that he repeat his work on new specimens and make duplicates available.

Mr. Traxler presented two papers at the February meeting of Association of Asphalt Petroleum Technologists in San Francisco advancing general rules and conclusions on similar phenomena and obtained by similar reasoning and the same type of evidence. When strong objections were raised from the floor, Traxler agreed with the validity of the objections and the desirability of more accurate reporting. The paper discussed above reveals that the previous discussions were of no avail. I regret to have to offer the above severe criticism of the paper, but it appears imperative in the interest of promoting accuracy in scientific publications that the statements made by the author of the paper be strongly refuted and the errors pointed out.

R. N. TRAXLER (*author's closure by letter*).—I disagree with Rostler's statement that the effects of oxidation, photooxidation, and volatilization are not evaluated by the procedures mentioned in the paper. I do not know when and where measurements of these effects have been made that are more to the point than those I have described.

Rostler's statement that component analysis is at variance with all other methods of analysis which are based on scientific principles of fractionation is a slanted statement. This procedure may be at variance with other methods based on solvent fractionation, but there is no ground for saying that these other unidentified procedures are based on more scientific principles of fractionation. The purpose of his comments is to discredit a component analysis procedure which has been proved of value in numerous practical applications. Criticism has been levelled at this component analysis procedure since the method was proposed a decade ago. However, Rostler's pronouncement that the method has been repeatedly proven false is a most unwarranted statement.

There is no basis for his statement that subtraction of "asphaltene" content from "asphaltic" (1-butanol insoluble) content results in some cases in a negative value for per cent resins.

The author's Coefficient of Dispersion $(R + C)/(A + S)$ was first presented at the asphalt symposium held in connection with the 138th National Meeting of the American Chemical Society, September 11–16, 1960, in New York City. This paper in a modified form was released for presentation at the Annual Meeting of the Association of Asphalt Paving Technologists in Charleston, S. C., February, 1961. In 1962, Rostler and White followed with their Durability Rating $(N + A_1)/(P + A_2)$ at the Annual Meeting of American Association of Paving Technologists in New Orleans, La.

The real cause for Rostler's attack may be our inability to correlate any arrangement of the fractions obtained by his sulfuric acid digestion procedure with the hardening of an asphalt when heated in air.

In the above discussion Rostler followed the practice employed by some reporters of quoting out of context, thereby conveying a meaning not found in the original and thus putting words into the author's mouth. For example, he states that erroneous definitions are given for oxidation, photooxidation, etc. These terms were given as descriptive names for particular test procedures and not as definitions.

The asphalts investigated were manufactured by well-known and ethical producers who are rightfully proud of the asphalt they have supplied for many years. Each asphalt is identified by a code number which is given to the company responsible for its production.

A FALLING PLUNGER VISCOMETER FOR DETERMINING ASPHALT VISCOSITY AT LOW TEMPERATURES

By R. J. Schmidt[1] and L. E. Santucci[1]

Synopsis

A falling plunger viscometer for determining the viscosity of paving grade asphalts in absolute units is described. The instrument can be used in most conventional, constant-temperature water baths. The unit is portable and simple to operate. Thus, a rapid determination of viscosity values results. The estimated cost of the viscometer is less than $500.

The repeatability of the data obtained from the falling plunger viscometer is examined at temperatures of 39.2 and 77 F for an 85–100 penetration grade Venezuelan asphalt. Data are also presented for a 200–300 Venezuelan asphalt and a 40–50, an 85–100, and a 200–300 penetration grade Californian asphalt.

The falling plunger viscometer is suggested as a simple, routine method for measuring asphalt viscosity and elastic properties at low temperatures. These qualities will become increasingly important in future specifications for paving asphalts.

"The methods commonly used for evaluating the susceptibility of a bituminous material, that is, the change of consistency with change in temperature, are dependent on arbitrary tests such as penetration, softening point, or time of efflux at an elevated temperature. Although consistency as measured by all of these empirical tests is some function of viscosity expressed in terms of absolute units, the relationship is frequently complicated as has been shown for penetration (**1,2**).[2] Since viscosity is a fundamental property of all fluid materials, the changes in consistency brought about by changes in temperature should be most satisfactorily expressed in terms of absolute viscosity units (**3**)."

This statement, written as an introduction in 1936 to a paper on asphalt viscosity by Schweyer, Coombs, and Traxler, shows the need for asphalt viscosity to be expressed in absolute units. The need is becoming more urgent as it becomes more evident that the properties of asphalt concrete are dependent on the rheology of the asphalt (**4–6**). The rates at which asphalt is sheared in asphalt concrete must vary over many orders of magnitude depending on the precise location under consideration between the rock particles, the geometry of the structure, and the loading conditions. Fundamental considerations of the system using an appropriate integration of the asphalt viscosity and shear rate will be an essential part of advanced asphalt concrete engineering designs. A contribution toward this objective will be to describe the properties of the components in fundamental terms and at clearly defined shear rates rather than in relative terms at unknown and varied shear rates.

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[1] California Research Corp., Richmond, Calif.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

Background

Neppe (2) has reviewed and analyzed a number of proposals intended to permit conversion of penetration, softening point, and other empirical bituminous tests into fundamental units. These widely used ASTM tests do not give values that can be used easily because they measure viscosity at unknown and varying stresses which change at different rates (3,7). For example, bitumens of the same penetration do not all have the same viscosity (1,8–11). In our own laboratory, as in any other major asphalt laboratory, these differences have been substantiated.

Efforts are now being made to reach this objective. Paving asphalt viscosities determined by capillary measurements at 275 and 140 F expressed in fundamental units are in the process of being adopted (12–14). These measurements will be most helpful to future understanding and specification of asphalt qualities at hot mixing temperatures and at temperatures representative of those obtained under road surface conditions on a hot day. Fundamental viscosity at surface temperatures has been shown to be related to the degree of pavement rutting obtained under traffic (15). Similar studies on the relation of rheology of asphalt at lower temperatures to the quality of asphalt concrete are being made and have demonstrated the importance of the use of fundamental units as a measure of asphalt quality (4).

Instruments to measure asphalt viscosity in fundamental units have been available for many years. Some of these methods (3,16) (capillary) are strictly fundamental only at temperatures where the asphalts approach becoming Newtonian—that is, the viscosity is not shear-dependent. Usually paving asphalts lose most of their shear dependency above 140 F. At lower temperatures, the asphalt viscosities usually become increasingly shear-dependent and as a result the nonlinear flow in the capillary tubes becomes more exaggerated. Variable and indeterminate shear rates exist when viscosity is determined under these conditions. Such values cannot be used satisfactorily in fundamentally based engineering studies, especially where dynamic considerations are prevalent.

Other test methods in which shear rates are reasonably uniform throughout the asphalt have been successful. One of these is the Couette-type rotating concentric cylinder viscometer (17) which was used extensively by Saal and Koens (18) to investigate the flow properties of asphalt. Mooney and Ewart (19) improved the instrument by making the ends of the cylinder conical in shape and thus maintaining a more uniform shear rate at the ends of the cylinder. Traxler and Schweyer (20) also investigated the use of this modified instrument with asphalt. A recent type of rotating instrument which subjects the material to a uniform shear rate is the Ferranti-Shirley cone-and-plate-type viscometer (21). In this instrument, the angle of the cone is made so that the velocity increase from the center to the outer edge of the cone is exactly offset by the increasing thickness of the film between the cone and plate.

The most recent and successful method proposed for use with asphalts is the sliding plate microviscometer, proposed originally by Labout and van Oort (22) and developed into a more practical instrument by Griffin and associates (23,24). Winniford has used this instrument to investigate the influence of asphalt composition on rheology (25) and the influence of filler on the rheology of the mixture (26). The microviscometer method complies with all of the classical requirements for determining viscosity. The shear stress is uniform and known. Furthermore, the method requires an exceptionally small amount of material (10 mg) and accordingly is useful for determining the viscosity of micro samples resulting from thin film exposure tests (23). Evans and Griffin (27) have modified the instrument so it is useful at low temperatures.

The simplest of the viscometers which maintain uniform shear rate is the falling plunger viscometer, first described by Segel (28) and used by Pochettino (29) for measuring the viscosity of pitch and by Mack (30) for the viscosity of asphalts. Extensive investigations of this instrument were made by Traxler and Schweyer (20) who demonstrated that it could be used to measure the viscosity of asphalts from 10^3 to 10^9 poises.

Other than the capillary instruments, most of the instruments discussed were intended and used for research purposes. Their original cost and the time required for each determination precluded their use as specification methods. An exception to this was Traxler's (20) falling plunger viscometer which was proposed in 1936 for specification purposes. Evidently the need to specify asphalts in fundamental units was not imperative enough at that time to stimulate adoption of his excellent method. More recently, the microviscometer has been proposed for this purpose and to some extent has been used successfully. For example, in some of the newest specifications proposed, Hveem (14) includes microviscometer values on laboratory-aged specimens.

At temperatures above about 140 F, the capillary instruments appear to be adequate to describe the viscous properties of asphalts. Complex viscoelastic properties become increasingly important as the asphalt drops below this temperature. It is at these lower temperatures, occurring during the thawing of frozen pavements when the base is least stable, that a need exists for an adequate instrument and method. Brown and Sparks (31) have reported that elastic properties of asphalt are especially significant at low temperatures. Of the methods proposed, only the modified microviscometer described by Evans and Griffin (27) seems useful at the low temperatures.

REQUIREMENTS FOR AN ACCEPTABLE LOW-TEMPERATURE VISCOMETER

A test method and instrument should meet the following requirements if it is to be adopted widely for specification purposes:

1. The time at which the method is proposed must be right; that is, there should exist a strong desire by the users and producers for an appropriate test.
2. The equipment should be relatively inexpensive.
3. The instrument should be rugged enough to resist damage during routine use in the laboratory.
4. The instrument operation and the viscosity calculations should be simple enough so that technicians can satisfactorily and routinely perform the tests.
5. One technician should be able to perform a large number of determinations in a day.
6. For control purposes, the elapsed time for each determination should be as short as possible.
7. The known and uniform shear stress should be applied simultaneously to all parts of the asphalt so that results expressed in absolute units are valid.
8. It should be possible to vary the shear stress over a wide range and also to measure the elastic effects so that the rheology of the asphalts can be observed and considered in pavement studies.
9. The method should be insensitive

to an occasional foreign solid particle included in the asphalt.

10. The instrument should be capable of determining asphalt viscosities at near freezing temperatures where cracking and freeze-thaw pavement failures are likely to occur.

11. The test results should be reproducible and repeatable.

The falling plunger viscometer method proposed in this paper complies reasonably well with these requirements. As expressed by Hveem (14), the need for an appropriate test now exists. The instrument cost is low; moreover, it is rugged and simple to operate. Most important, at least twelve determinations in an 8-hr day can be made at 77 F by one technician equipped with two instruments along with two extra cylinders and plungers. At 39.2 F the rate may be reduced to eight a day. The elapsed time to test an asphalt at 39.2 F is about

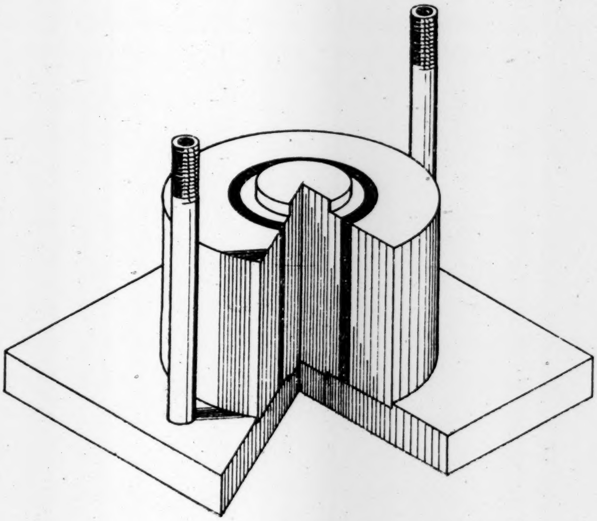
FIG. 1.—Asphalt Viscometer Mold Assembly.

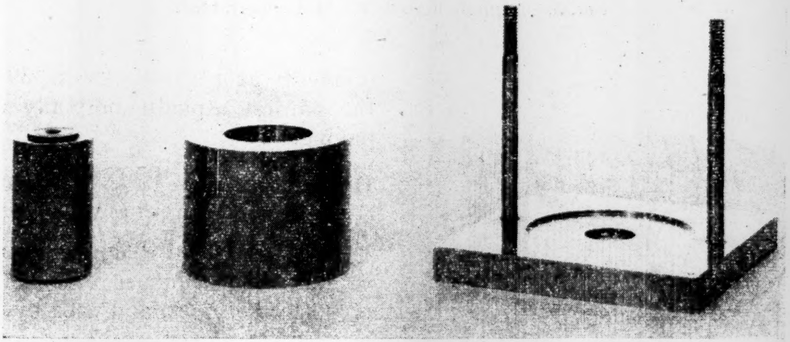
FIG. 2.—Asphalt Viscometer Mold Disassembled.

3½ hr, which is a short enough time for control purposes. The design of the instrument assures that all parts of the asphalt under test are subjected to the same shear stress. In addition, the stress can be varied over a wide range. Also, the film of asphalt tested is thick enough so that the sample to be tested can be strained free of debris large enough to be a problem. Most important is the ability of the instrument to reveal and measure the transient elastic and viscous qualities usually not apparent in most other simple test methods. It provides measurements at least as low as 39.2 F on the hardest asphalts normally used in paving.

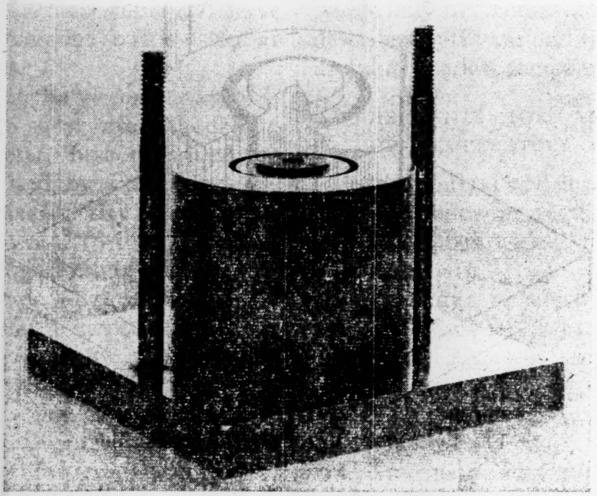

FIG. 3.—Asphalt Viscometer Mold Assembled.

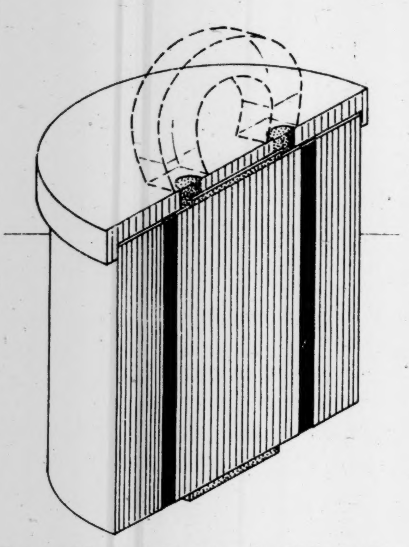

FIG. 4—Magnetic Retainer for Cylinder-Plunger Alignment.

DESCRIPTION AND TEST PROCEDURE FOR FALLING PLUNGER VISCOMETER

The instrument[3] is similar in principle to the falling plunger viscometer proposed by Segel (**28**) and used by Pochettino (**29**), Mack (**30**), and Traxler (**20**).

The principle of the instrument is illustrated in Figs. 1 to 7. Molten asphalt is poured into the space between the preheated inner plunger and outer cylinder. The annular space for the asphalt sample between the plunger and cylinder is 0.2 cm wide while the length of the

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[3] Construction drawings of the proposed falling plunger viscometer may be obtained from the authors.

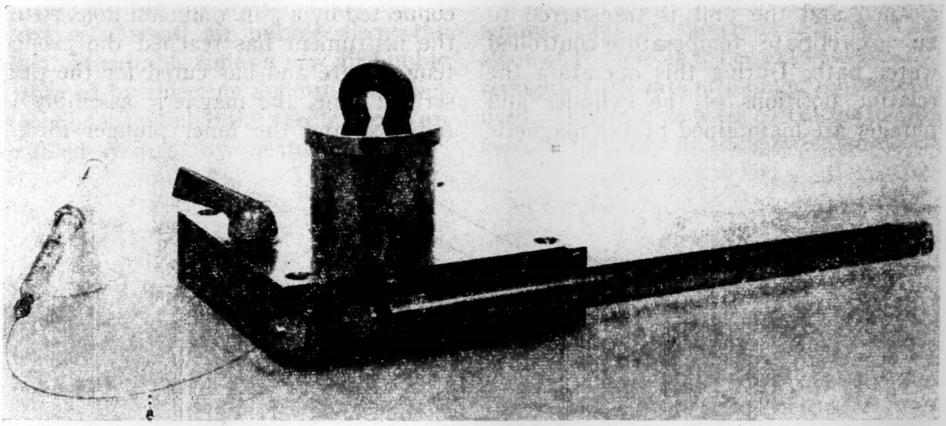

FIG. 5.—Magnetic Retainer Assembled.

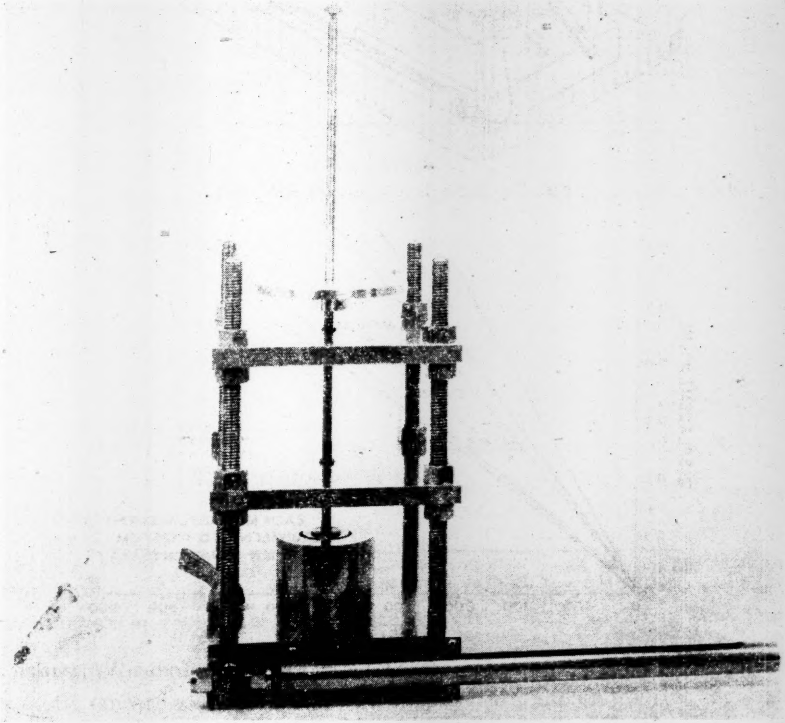

FIG. 6—Assembled Viscometer Ready for Operation.

space is 5.0 cm. The average total area of asphalt being sheared at the beginning of the test is 50.0 sq cm. The asphalt is shown in place in Fig. 1. The disassembled unit and the unit in an assembled form are shown in Figs. 2 and 3.

When the asphalt has cooled sufficiently, the ends of the plunger are cleaned and the unit is transferred to an appropriate temperature-controlled water bath. During this operation the relative positions of the cylinder and plunger are maintained by the magnetic assembly shown in Figs. 4 and 5. This assembly is shown in place on the base sample holder in Fig. 5. The outer cylinder rests on and is sealed with stopcock grease to a $\frac{7}{16}$-in. wide lip near the top of a circular cavity in the base holder. The cavity and a measuring pipet are connected by a $\frac{1}{8}$-in. diameter hole. After the instrument has reached the proper temperature and has cured for the prescribed time, the magnetic assembly is removed and the inner plunger forced downward into the water-filled cavity by means of applied weights. This step is illustrated in Figs. 6 and 7.

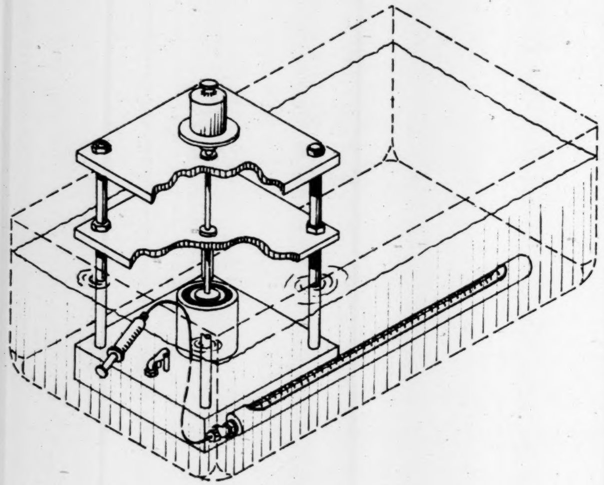

FIG. 7.—Asphalt Viscometer Assembly.

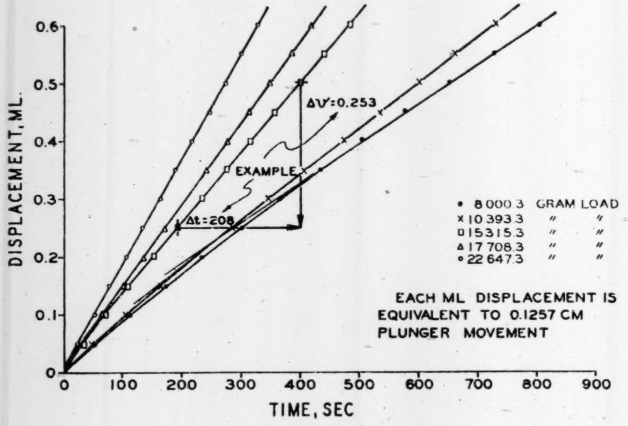

FIG. 8.—Viscometer Plunger Movement at 39.2 F for an 85–100 Penetration Venezuelan Asphalt.

The plunger movement is followed by measuring, in the small pipet shown in Figs. 6 and 7, the water displaced as the plunger falls. The water displaced can be followed readily by watching the movement of a small air bubble along the pipet. At suitable times a new air bubble is formed by injecting air into the pipet through a hypodermic needle from an air-filled syringe. By means of this hydraulic amplification, movements as small as 0.001 cm are evident. Although not essential for control in specification purposes, the movement can be sensed and recorded continuously. This latter step will eliminate the need to plot points to determine the rates of plunger movement.

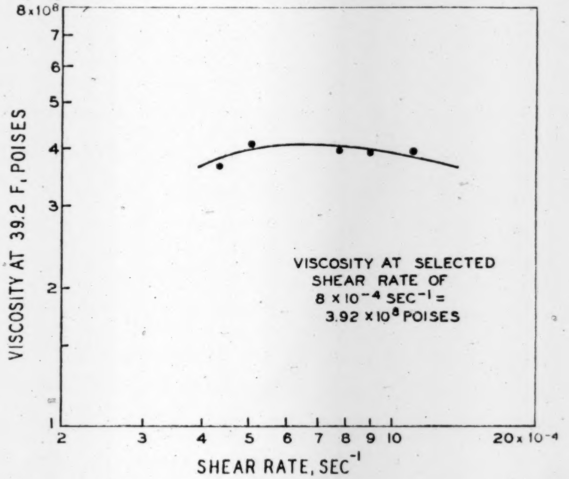

Fig. 9.—Relation of Viscosity to Shear Rate for an 85–100 Penetration Venezuelan Asphalt at 39.2 F.

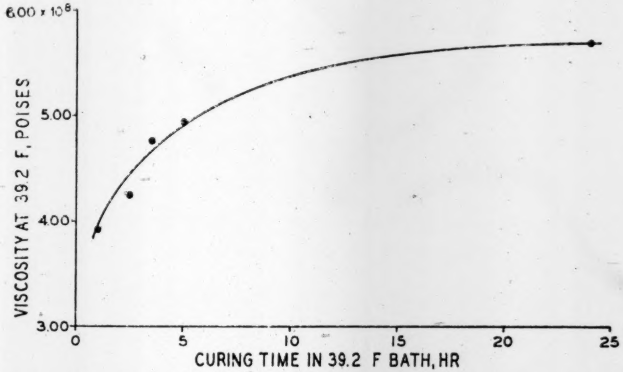

Fig. 10.—Increase in Viscosity of a Venezuelan Asphalt at 39.2 F as Curing Time Increases.

Movement of the plunger is plotted against time as shown in Fig. 8. The slope of the line as it becomes essentially straight is the rate of plunger movement used. This rate is used to calculate the asphalt viscosity at the particular shear rate selected. An example of viscosities found are shown plotted against shear rates in Fig. 9. Details of the procedure and calculations are given in the Appendix.[4]

Figure 10 illustrates the wide variations in viscosity that can be obtained by using long and short curing periods on the specimen in the bath before testing. This phenomenon is well known and is the basis for such careful control of curing periods in all asphalt testing. The effect becomes more pronounced as the testing temperature becomes lower. For this reason poorer precision is obtained in low-temperature measurements than is obtained at higher temperatures.

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[4] See p. 1260.

TABLE 1.—PRECISION OF VISCOSITY MEASUREMENTS AT 39.2 F FOR AN 85–100 PENETRATION VENEZUELAN ASPHALT.

Mean value, $\bar{x}$, poises = 3.92×10^8
Maximum value, poises = 4.56×10^8
Minimum value, poises = 3.35×10^8
Range, poises = 1.21×10^8
Standard deviation, s = 0.379
Coefficient of variation, per cent of mean = 9.7
Repeatability, 95 per cent confidence limits, per cent of mean = 29.4

Run	Viscosity,[a] poises
1	3.47×10^8
2	3.35
3	3.68
4	3.41
5	4.50
6	4.56
7	4.00
8	3.87
9	3.92
10	4.16
11	3.77
12	3.71
13	4.00
14	4.00
15	4.45

[a] Shear rate selected for reported viscosity results = 8×10^{-4} sec^{-1}.

The rate of change of viscosity with curing time will be different for each asphalt. Some will reach equilibrium in a few hours; others may take days or weeks to stop changing. True equilibrium can be assured only after a long curing time. This would be impractical for a routine test procedure; therefore a definite curing time was selected arbitrarily. The period selected is 1 hr, which is approximately the same as the interval required by the ASTM penetration test.[5] A shorter or longer curing period would change the elapsed time to run a test accordingly.

REPEATABILITY OF THE FALLING PLUNGER VISCOMETER

The data shown in Tables 1 and 2 are examples of the values obtained with our falling plunger viscometer using the procedure described in the Appendix.

TABLE 2.—PRECISION OF VISCOSITY MEASUREMENTS AT 77 F FOR AN 85–100 PENETRATION VENEZUELAN ASPHALT.

Mean value, $\bar{x}$, poises = 1.06×10^6
Maximum value, poises = 1.17×10^6
Minimum value, poises = 0.93×10^6
Range, poises = 0.24×10^6
Standard deviation, s = 0.0702
Coefficient of variation, per cent of mean = 6.6
Repeatability, 95 per cent confidence limits, per cent of mean = 19.8

Run	Viscosity,[a] poises
1	1.04×10^6
2	1.03
3	1.10
4	0.93
5	0.99
6	1.12
7	1.12
8	1.14
9	1.12
10	1.04
11	0.98
12	0.99
13	1.17
14	1.03
15	1.03
16	1.10
17	1.16

[a] Shear rate selected for reported viscosity results = 1×10^{-2} sec^{-1}.

These data were obtained by one technician using two complete units and two separate cylinders and plungers. All values were made by pushing the plunger in one direction. A typical distribution pattern of viscosity results obtained at 39.2 F on an 85–100 penetration Venezuelan asphalt is given in Fig. 11. The

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[5] Method of Test for Penetration of Bituminous Materials (D 5), 1961 Book of ASTM Standards, Part 4.

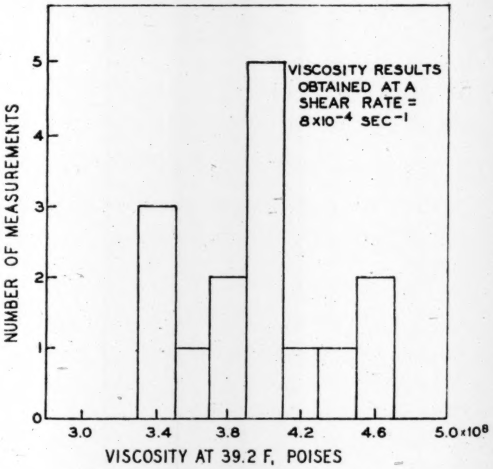

Fig. 11.—Distribution Pattern for Viscosities at 39.2 F of an 85–100 Penetration Venezuelan Asphalt.

standard deviation, coefficient of variation, and repeatability were calculated using recommended practices for applying precision data as given by ASTM for petroleum products and lubricants (32). The repeatability at 39.2 F is shown to be 29.4 per cent with a coefficient of variation of 9.7 per cent. At 77 F a repeatability of 19.8 per cent and a coefficient of variation of 6.6 per cent were obtained.

Additional viscosity data were obtained at 39.2 F on a 200–300 penetration Venezuelan asphalt and a 40–50, an 85–100, and a 200–300 penetration Californian asphalt. These results, along with their coefficients of variation, are shown in Table 3.

TABLE 3.—PRECISION OF VISCOSITY MEASUREMENTS AT 39.2 F FOR VARIOUS PENETRATION GRADE ASPHALTS.

Asphalt Penetration and Source	Number of Technicians	Number of Tests	Mean Viscosity,[a] poises	Coefficient of Variation, per cent of mean
200–300, Venezuela....................	1	9	0.88×10^8	9.4
200–300, California....................	1	7	0.37	13.7
85–100, California....................	1	5	6.68	6.7
85–100, Venezuela....................	1	14	3.92	9.7
40–50, California....................	1	6	27.4	12.2

[a] Shear rate selected for reported viscosity results = 8×10^{-4} sec^{-1}.

Discussion

The pattern of viscosity change with shear rate shown on Fig. 9 was found to be common at the low shear rates used. This effect is evident at both 77 F and 39.2 F. As shown typically in Fig. 9, the viscosity rises to a maximum as the shear rate increases and then declines in the way usually associated with non-Newtonian liquids. This type of behavior is not unknown. A common example would be concentrated suspensions of rigid particles such as wet beach sand.

A possible mechanism for this phenomenon is that if enough time is allowed the entanglements or interactions between the constituents responsible for shear-susceptible viscous resistance relax, reform, and accommodate movement without contributing to the viscous resistance. At high shear rates, these entanglements and associations are destroyed faster than they can form in the viscous system. A maximum viscosity will occur somewhere between these extremes.

This shear susceptible viscous (and elastic) resistance will approach an equilibrium value following the application of a steady shear rate for a long period of time. Under steady state conditions, at each new shear rate the amount of viscous resistance may be different. Winniford (25) demonstrates the rate of buildup and destruction of "asphalt structure" which we have attributed to associations and entanglements between asphalt components. For convenience, we have chosen to define these temperature-time-shear history dependent viscous and elastic elements as transient viscoelastic elements.

These peculiarities are precisely the type of phenomenon that must be shown and measured by a test method if it is eventually to allow meaningful specifications to be developed. We are of the opinion that the dependency of the viscosity and the elasticity on the temperature-time-shear rate history of the asphalt (transient viscoelastic properties) is responsible for most of the anomalies in asphalt behavior and testing. We expect that establishment of an exact sequence of events through the temperature-time-shear rate history of the asphalt will result ultimately in a reproducible and meaningful specification test method. The detailed procedure in the Appendix is a step in this direction.

The procedure is arranged presently to be sensitive to the transient viscoelastic properties which become especially large at low temperatures. Variations in time and shear rate history now permitted by the procedure cause larger variations in the repeatability than desired. Suitable modification and precise standardization of the procedure are expected to reduce the variance to an acceptable value.

In the procedure used by Griffin and associates (23,24) and Evans and Griffin (27) with the sliding plate microviscometer, the specimens are subjected to the highest shear rates first. This procedure destroys most of the transient viscoelastic properties formed during the time the specimens have been allowed to stand at constant temperature before testing. As a result of this initial high shear exposure, they sense mostly the basic viscous qualities and minimize the transient viscoelastic qualities of the asphalt. Very little of these changeable properties are shown by their method. In our opinion, these changeable properties will be found to be most significant in paving asphalts.

SUMMARY

A falling plunger viscometer is proposed as an instrument for eventual use as a specification testing device. The method is rapid and the instrument is low in cost, rugged, and simple to operate. At this early stage of development the precision is reasonably good, especially when the high degree of transient viscoelastic properties retained during the test is considered. Further development of the method and instrument is expected to increase the precision substantially. The viscometer determines the viscous properties in a classically correct manner and accordingly allows viscosity measurements to be made at temperatures at least as low as 39.2 F on a 40–50 penetration asphalt.

Acknowledgments:

The authors acknowledge the support of the American Bitumuls and Asphalt Co. who sponsored this work.

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APPENDIX

PROCEDURE FOR DETERMINING ASPHALT VISCOSITIES WITH FALLING PLUNGER VISCOMETER

Sample Preparation:

Heat a filled 3-oz penetration can sample of asphalt to 275 F in a controlled-temperature oven. The can should be tightly sealed while in the oven. Pour the asphalt through a No. 100 square mesh sieve to remove foreign particles. Pouring the asphalt should require a maximum time of 5 min. The times allotted for each step in the procedure should be strictly adhered to for good repeatability in the test results. If the technician completes a step before the allotted time, he should wait until this time has elapsed before proceeding to the next step.

Return the screened asphalt to a 275 F oven for 30 min. Allow the assembled mold base, Teflon Fiberglas gasket, cylinder, and plunger (Fig. 1) to preheat in a 275 F oven for 1 hr. Remove assembly and asphalt from the oven. Set assembly on an asbestos pad and fill the annular space between plunger and cylinder with the hot asphalt. Add enough extra asphalt into the space to provide for shrinkage on cooling. Fill from one position so the asphalt runs to and fills from the bottom. This is necessary to avoid air pockets. Allow to cool for 25 min.

Immerse the assembly in a room-temperature water bath until it has cooled sufficiently to handle with the hands. For soft asphalts (200–300 penetration), it may be necessary to immerse the specimen in an ice water bath to prevent flow of the asphalt from the annular space. This step is completed in 5 min.

With a heated knife, remove excess asphalt so it is even with the top of the plunger and cylinder. Set the sample cover on top of the plunger and cylinder. Set the small Alnico magnet so its tips cover the two circular iron inserts (Fig. 4). The magnetic flux now passes through the small inserts to the circular insert on the plunger. This magnetic assembly holds and prevents movement of the plunger with respect to the cylinder. Grasp the cylinder and lift the combined cylinder, plunger, and magnetic holder carefully from the mold base. Invert and remove the gasket, clean with a heated knife, and grease the cylinder face with stopcock grease. This step is completed in 10 min.

Assembly for Viscosity Determination:

The sample holder is placed on a firm base in the temperature-controlled water bath. The face in the holder matching the cylinder is lightly coated with stopcock grease. Transfer the cylinder, plunger, and magnetic assembly to the water bath and place it into position on the base. Twist the assembly to cause a tight fit. The valve in the base should be open to the water bath during this operation. As soon as this operation is completed, turn the valve, closing off the chamber in the base. The plunger is now supported both magnetically and hydraulically. The magnetic assembly can be removed at any convenient time by turning the magnet on the cover so the tips of the magnet no longer cover the two circular iron inserts. This breaks the magnetic flux, releases the plunger, and permits the sample cover and magnet to be removed. Five minutes is allowed for this part of the procedure.

Allow the sample to remain 1 hr in the water bath before proceeding with viscosity determinations. Place the rod extending from the bottom of the platform through the support guide onto the center hole drilled in the plunger. Add the desired weights to the platform. Add the smallest weight first.

NOTE.—The weights used in our test with a typical 85–100 penetration asphalt were approximately 8000, 10,400, 15,300, 17,700, and 22,650 g in the order of application. For other grades of asphalt tested at 39.2 F, the following weight range is suggested to bracket the selected shear rate of 8×10^{-4} sec^{-1}:

Penetration Grade	Weight, g
200–300	1 000 to 4 000
40–50	25 000 to 60 000

The shear rate of 8×10^{-4} sec^{-1} is selected for convenience only. This selection was based on the load limitations of the base plate of the water bath in which samples were tested.

Viscosity Determination:

When ready to proceed, inject a small air bubble into the pipet through a long hypodermic needle by means of an air-filled syringe. Next, turn the valve so the hydraulic chamber and pipet are connected and open to the water bath at the same time. The plunger is now able to slowly descend into the hydraulic chamber, thus forcing the air bubble toward the open end of the pipet. To avoid correction for a variable head of water, the pipet should be mounted horizontally near the bottom of the water bath at the same level as the hydraulic chamber.

Start the stop watch as the meniscus of the air bubble passes the first convenient mark on the pipet. Record the time as it passes each convenient mark. When sufficient readings have been taken to assure a steady rate, turn the valve so the chamber is closed off. Add more weight to the platform and inject a new air bubble into the pipet with the syringe and hypodermic needle. Repeat as before but at an increased shear rate. When more than 0.40 cm total movement (8 per cent) of the plunger is obtained, reverse the cylinder and plunger on the sample holder and proceed. This forces the plunger in the reverse direction with respect to the cylinder.

NOTE.—We have found reversal of the plunger and cylinder is not necessary at 39.2 F. A plunger movement of 0.08 cm is allowed for each applied load. When reversal is carried out at this temperature, the log viscosity-log shear rate plot shows a decrease in viscosity with increasing shear rate to a minimum, followed by an increase in viscosity with further increasing shear rate. The normal viscosity-shear rate relationship shows an increase in viscosity to a maximum followed by a decline. This change in the shape of the log viscosity-log shear rate plot is attributed to the elasticity effect in the asphalt. The unknown elastic contribution to the shear of the asphalt film after inversion of the plunger results in a low viscosity determination. At 77 F and higher testing temperatures where reversal of the plunger and cylinder may be required, only infrequent changes in the log viscosity-log shear rate plot were noted. This is probably due to the shorter time required at these temperatures to dissipate the small elastic component in the asphalt.

Calculations:

Viscosity, η

$$= \frac{\text{Force per unit area (dynes)}}{\text{Rate of movement per unit thickness (cm per sec per cm)}}$$

$$= \frac{(\text{Load in grams} \times 980) / (\text{Area in sq cm})}{(\text{Movement in cm per sec}) \times (1/\text{Film thickness in cm})} = \frac{S_0}{dv/dr}$$

$$\text{Area} = \text{Height of cylinder} \times \frac{\text{Circumference of cylinder} + \text{circumference of plunger}}{2}$$

$$= 5.0 \times \frac{(10.629 + 9.371)}{2} = 50.0 \text{ sq cm}$$

Load = Platform weight + weights added + plunger weight in water.

Rate = dv/dr = Measured from water movement in pipet where 1-ml displacement = 0.1257-cm plunger movement. Rate determined by plotting time versus movement and drawing smooth curve through points.

Thickness = Radius of cylinder − radius of plunger = 0.2 cm

$$S_0 = \frac{\text{Load} \times 980}{\text{sq cm area}}$$

Examples of Calculations (From Figs. 8 and 9):

Test temperature = 39.2 F

Asphalt = 85–100 penetration Venezuelan asphalt

Area of asphalt sheared = 50 sq cm

Thickness = 0.2 cm

Load = Platform (252.7 g) + added weights (14,797.2 g) + plunger weight in water (265.4 g) = 15,315.3 g

Rate = 0.253 ml in 208 sec (from plot of points, Fig. 8) equal to 0.253 × 0.1257 = 0.0318 cm per 208 sec

$$\frac{dv}{dr} = \frac{0.0318}{208 \times 0.2}$$

$$= 7.644 \times 10^{-4} \text{ cm per sec per cm}$$

$$S_0 = \frac{15{,}315.3 \times 980}{50}$$

$$= 3.002 \times 10^{5} \text{ dynes per sq cm}$$

$$\eta = \frac{S_0}{dv/dr} = \frac{3.002 \times 10^{5}}{7.644 \times 10^{-4}}$$

$$= 3.93 \times 10^{8} \text{ poises}$$

DISCUSSION

MR. H. E. SCHWEYER.[1]—Do the authors have any comparable data for the sliding plate viscometer and the falling plunger viscometer?

MESSRS. R. J. SCHMIDT AND L. E. SANTUCCI (*authors*).—To the authors' knowledge, no comparable data are available for the sliding plate viscometer and the falling plunger unit in the same shear rate range.

MR. F. S. SCOTT[2] (*presented in written form*).—The authors gave us the opportunity several months ago of constructing the laboratory equipment described in their paper. The equipment is simple to construct and does in fact meet the criteria for specification uses listed in the paper. We have tested a fairly wide variety of asphalts at temperatures between 39.2 F and 95 F and have noted that viscosity does rise to a maximum as the shear rate increases and then declines at higher shear rates. The laboratory equipment will be very useful in research studies of the "transient viscoelastic elements" which the authors appear to have uncovered. In regard to its use as a control instrument, we believe the authors are rather optimistic in their statement that eight determinations per day can be made at 39.2 F by a single operator using the procedure outlined in the paper. Work is under way in our laboratory, and perhaps in other laboratories, to reduce testing effort without seriously affecting the usefulness of the test results for routine specification purposes.

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[2] Union Oil Co. of California, Research Center, Brea, Calif.

RHEOLOGICAL PROPERTIES OF ASPHALT-AGGREGATE COMPOSITIONS*

By E. M. Krokosky,[1] E. Tons,[1] and R. D. Andrews[1]

Synopsis

The rheological properties of some typical asphalt - crushed rock systems in the composition range commonly used in road surfacing (approximately 15 per cent asphalt by volume) have been investigated. These materials have been studied by compression creep and recovery, compression stress-strain measurements, and stress-relaxation.

Asphalt itself has been found to be a simple viscoelastic material. However, when large amounts of inert filler are added, the composite system shows nonlinear and pseudoviscoelastic behavior. The possibility of separating the nonlinear (strain and stress) effects from time and temperature effects was investigated. Time-temperature superposition was attempted, and shift factors compared with those for pure asphalt.

The rheological behavior is found to depend on the type of test employed, since interparticle interactions are being observed to a different extent in the different types of test. These interactions (which are not time-dependent) are observed most strongly in stress-strain data, and least in stress-relaxation data. The normal interconvertibility of different types of viscoelastic data is therefore not observed in systems of this type; a proper characterization of their rheological properties therefore cannot be obtained by a single type of test, but only from a combination of tests.

Notations

AC = Asphalt content

d/dt = Differentiation with respect to time

D_I = Instantaneous compliance

D_R = Recovery compliance

$D(t)$ = Compliance in creep

$E(t)$ = Stress relaxation modulus

ϵ = Strain

$\dot{\epsilon}$ = Strain rate

η_c = Compression viscosity

η_s = Shear viscosity

$F(t)$ = Constant rate of strain modulus

$\Gamma(\epsilon)$ = Factor expressing deviation from linear behavior in stress relaxation

$\Gamma^*(\sigma)$ = Factor expressing deviation from linear behavior in creep

$J(t)$ = Linear creep compliance

m = Factor relating $F(t)$ to $E(t) \equiv (d \log F(t))/(d \log t)$

ρ = Density

$S(t)$ = Linear constant rate of strain modulus

σ = Stress

t = Time

T = Temperature

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* Presented at the Sixty-sixth Annual Meeting of the Society, June 23–28, 1963.

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The rheological behavior of materials that obey the principles of linear viscoelasticity, such as amorphous polymers, has been studied in considerable detail during the past decade or two, and can now be clearly described and formulated. However, the deformation and flow characteristics of highly filled viscoelastic materials are not well understood at the present time, and as yet no adequate models exist to represent their behavior. In the present investigation, an experimental study was carried out on a composite system consisting of asphalt and crushed rock aggregate (bituminous concrete) to determine the extent to which the rheological behavior of this system can be described by, or related to, simple viscoelasticity theory. Such information is of importance in pavement design, but is also of broader theoretical interest as representing a prototype composite system of the sort mentioned above, and the results should be helpful in advancing the understanding of such related systems as solid rocket propellants and water-filled soils.

Pure asphalt is known to be a linear viscoelastic material. It might be suspected, therefore, that a composite system of asphalt-aggregate could be represented by a model of the ordinary viscoelastic type, consisting of elastic springs and viscous dashpots in some sort of combination, which would show a linear response (stress and strain proportional). Initial consideration of the asphalt-aggregate system would suggest that such a treatment might be possible, since one of the two phases (the asphalt) is much more fluid than the other, and the deformation of the composite might consequently be expected to be localized entirely in the asphalt phase. The aggregate particles will undoubtedly show no deformation in themselves, and might then act simply as an inert filler; they would affect the deformation behavior of the composite only by some geometrical rearrangement as the asphalt phase deforms.

The viscoelastic properties of pure asphalt have been studied in some detail, using the techniques employed in polymer research (**1–4**);[2] this work has been summarized in reference (**5**). More recent work by Wada and Hirose (**4**) showed that the temperature-dependence of the retardation times obeys the Williams-Landel-Ferry (WLF) equation. For typical polymers, the standard temperature, T_s, in the early form of the WLF equation is approximately $T_s + 50$; for the asphalts it was found to be equal to $T_s + 56$. The shift factors, a_T, obtained by Wada and Hirose from horizontal shifting of their torsional creep data were in good agreement with the WLF equation. They also showed that a_T was related to the steady-state flow viscosity of the asphalt by the normal relation, $a_T = \eta/\eta_0$.

The viscoelastic properties of asphalts may be summarized as follows:

1. Asphalts above their glass transition temperature, T_g, are viscoelastic liquids with characteristics quite similar to polymer solutions.
2. Modulus versus log frequency curves lack a plateau region, indicating that there are no cross-links or molecular entanglements present in asphalts.
3. The principle of time-temperature superposition is applicable to asphalts.
4. The effect of temperature is similar to that observed for linear polymers, and the WLF equation is obeyed over a wide range of temperature.

Earlier attempts to represent the behavior of asphalt-aggregate composites by simple viscoelastic models, consisting of a small number of springs and dashpots, were not completely successful

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

(6,7). The state of development of such models as of June, 1962, has been reviewed in detail elsewhere (5) by one of the present authors.

In order to describe the rheological properties of bituminous concrete in a more adequate way, the most obvious first step was to use a representation corresponding to an infinite or generalized mechanical model rather than a model consisting of a finite number of elastic and viscous elements. By use of such a model with a continuous spectrum of retardation or relaxation times, any time function obtained from a single type of experiment can be fit exactly. This type of description represents the most general formulation of linear viscoelastic behavior.

One of the important features of linear viscoelastic behavior is that it is possible to predict the results of one type of viscoelastic experiment from data for another type of experiment. Therefore, in the present investigation, three different types of experiment were carried out on the composites: creep, stress relaxation, and stress-strain measurements at constant rate of strain. It is essential to check this interconvertibility, in deciding whether a system can be correctly described as viscoelastic. Data from a single type of experiment can always be fit by the mathematical model, as indicated above, and the ability to do this for a single type of experiment therefore does not provide any real test of the nature of the system.

It was found that even this most general form of linear viscoelastic representation did not provide an adequate description of the behavior of the composite system. A still broader formulation can be provided by allowing the viscoelastic behavior to be *nonlinear*. This complicates the representation, and could lead to situations in which the mechanical model (which could contain nonlinear springs or nonlinear dashpots, or both) would no longer be unique. The theory in this area is largely undeveloped, and presents some difficulties in its most general form. A simple form of nonlinearity would be that in which strain and stress were no longer proportional, but the spectrum of relaxation or retardation times remained undisturbed (independent of stress or strain). This behavior seems to be observed in rubbery polymers at higher strains, and has been discussed by Guth, Wack, and Anthony (8), who showed that the stress could be factored into independent strain and time functions:

$$\sigma(\epsilon, t) = \Gamma(\epsilon)E(t) \ldots\ldots\ldots\ldots (1)$$

Leaderman (9) and Smith (10) have treated such systems.

In treating our own data, we have similarly introduced a function, Γ, as a multiplying factor for the stress or modulus, to represent deviations from apparent linear viscoelasticity. However, in contrast to the case above, where Γ is regarded as a function of strain alone, we have allowed our non-linearity function to be a very general function of strain, time, and temperature. No assumptions are made in advance as to its fundamental significance.

From a standpoint of still greater generality, rheological behavior which was not, or was not completely, viscoelastic in nature would also be possible. (The term "viscoelastic" indicates a combination of viscosity and elasticity, with no other effects present, while the term "rheological" simply indicates deformation or flow, without any implication as to the nature of the processes involved.) If the filler particles contributed any mechanical effect of their own to the over-all rheological behavior of the composite (that is, if they were not acting as a strictly "inert" filler), which is clearly possible, the system could no longer be described as simply viscoelastic. Since such a system might show rheological time effects in a single type of experiment which were indistinguishable from the behavior observed in a linear or nonlinear viscoelastic system, the term "pseudoviscoelastic" might be an appropriate description for such a system.

Viscoelastic and rheological descriptions are phenomenological in nature. The ultimate test of their applicability is, therefore, provided by an examination of the nature of the experimental behavior of the system. A selection of the experimental data obtained on a set of asphalt-aggregate composites will be presented in the following section, and the characterization of the observed behavior in terms of the framework indicated above will be discussed.

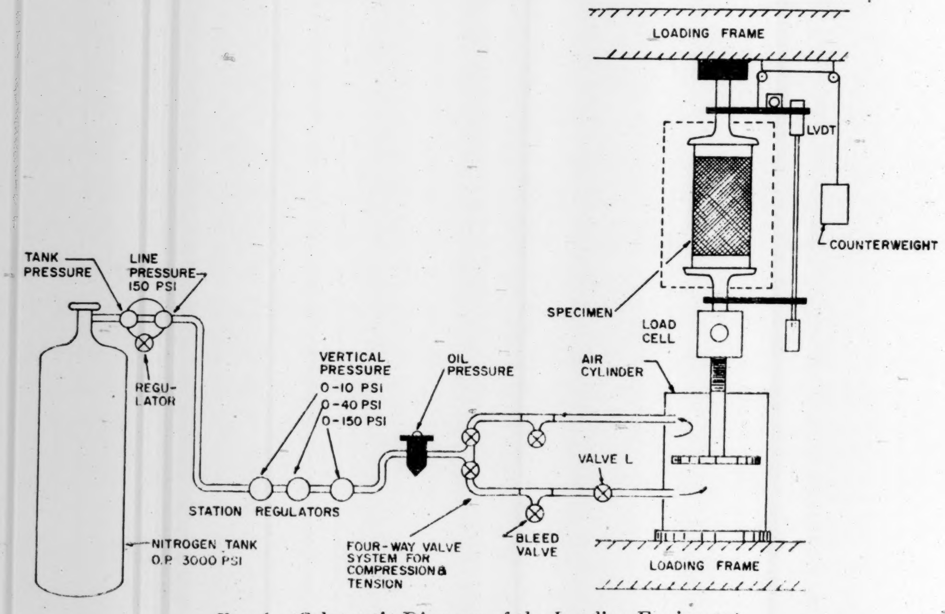
LOADING FRAME
LVDT
COUNTERWEIGHT
SPECIMEN
LOAD CELL
AIR CYLINDER
TANK PRESSURE
LINE PRESSURE 150 PSI
REGULATOR
VERTICAL PRESSURE
0-10 PSI
0-40 PSI
0-150 PSI
OIL PRESSURE
VALVE L
STATION REGULATORS
NITROGEN TANK O.P. 3000 PSI
FOUR-WAY VALVE SYSTEM FOR COMPRESSION & TENSION
BLEED VALVE
LOADING FRAME

FIG. 1.—Schematic Diagram of the Loading Equipment.

EXPERIMENTAL PROCEDURES

Equipment:

For the creep experiments it was necessary to construct a loading apparatus that could maintain a constant compressive force for a long period of time. A nitrogen pressure system was developed which maintained constant pressure through a series of regulators (Fig. 1). The accuracy and the constancy of the load was ascertained by means of a strain gage load cell and a proving ring. The deformation of the specimens under load was measured by a linear variable differential transformer (LVDT). This was used in combination with a commercial unit containing input, amplifier, and read-out device. The output from this unit was fed into a 12-in. width chart recorder. This provided a continuous record of the deformation as a function of time.

The constant rate of compression tests and the relaxation tests were conducted on an Instron testing machine, permitting rates of deformation from 0.02 in. per min to 20 in. per min. The load was registered on the standard Instron recorder and since the rate of specimen deformation and the chart speed were known, it was possible to read the amount of deformation also from the chart on which the load was being recorded.

Temperature regulation was achieved by placing an insulated box around the loading frame as illustrated in Fig. 2. Temperatures above room temperature were obtained using heating coils controlled by a bimetallic thermoregulator and relay, and rapid air circulation was maintained by two fans which were placed above the heating coils. Temperatures below room temperature were achieved by means of a dry ice cooling unit which forced cool air into the insulated box.

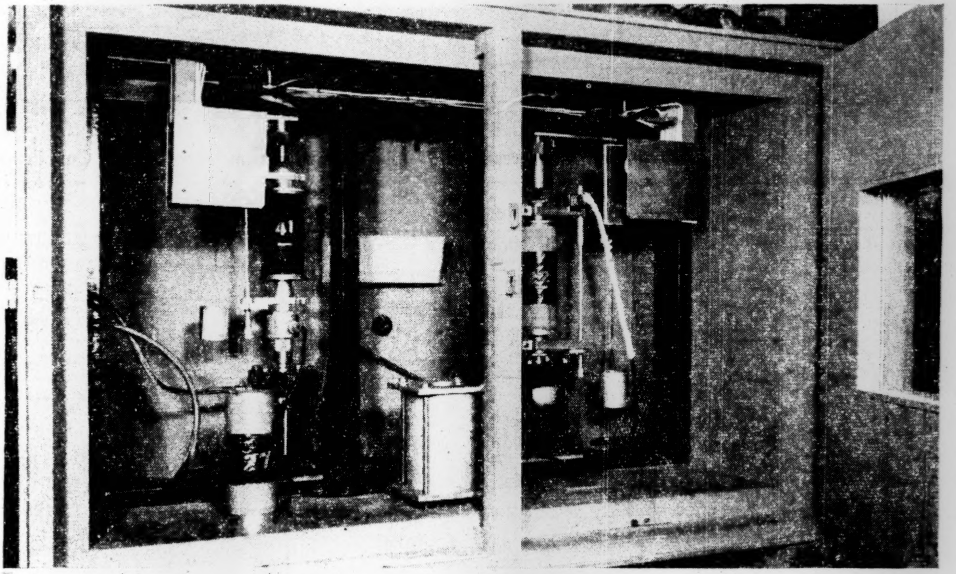

FIG. 2.—Creep Test Equipment.

For the constant rate of compression tests and relaxation tests on the Instron machine, temperature control was provided by an insulated chamber which was placed around the specimen, and which was connected directly to a small heating and cooling unit.

Specimen Preparation and Description:

In order to ensure maximum reproducibility of results, coarse and fine aggregate were separated by sieving into various fractions. The coarse and fine aggregate were then recombined in desired ratios and heated at 300 F for 8 hr. The asphalt at 300 F was added and the ingredients mixed for $1\frac{1}{2}$ min in a mechanical mixer and 1 min by hand. The specimens were then compacted statically for 1 min at 28,700 lb in a Smith triaxial specimen mold. The specimens were 3.82 in. in diameter and approximately 7.5 in. in length. Their average densities and void contents are given in the Appendix.[3] The ends of the specimens were ground off smoothly to ensure uniform loading and a true measure of deformation. In the creep tests, the

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[3] See p. 1285.

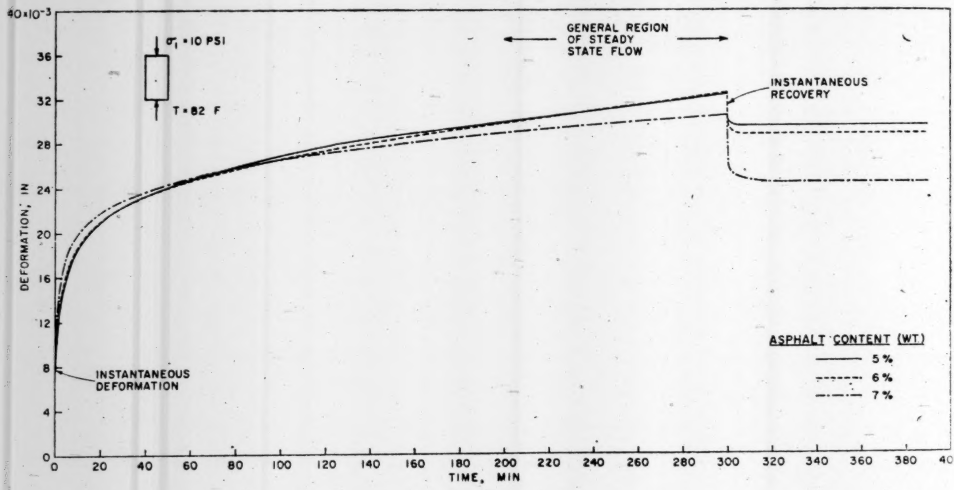


FIG. 3.—Creep Test (Unconfined Compression); Typical Deformation Versus Time Curves at 82 F for 5, 6, and 7 Per Cent Asphalt.

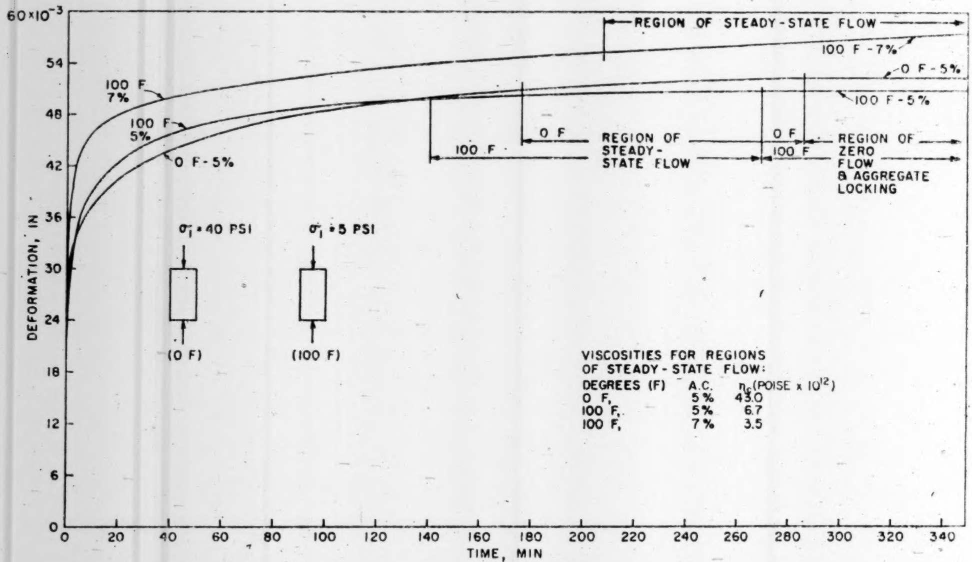


FIG. 4.—Creep Test (Unconfined Compression); Typical Deformation Versus Time Curves at 0 F and 100 F for 5 Per Cent Asphalt; at 100 F for 7 Per Cent Asphalt.

uniformity of loading was further enhanced by gluing aluminum caps on the polished ends of the specimens.

EXPERIMENTAL RESULTS

Creep and Creep Recovery:

Application of a constant load on an asphalt-aggregate composite for a period of time produces the type of deformation curve shown in Fig. 3 which is a replica of the creep curves as they appear on the recorder chart. Fig. 3 shows the deformation versus time curves for mixtures with the same applied load, temperature, dis-

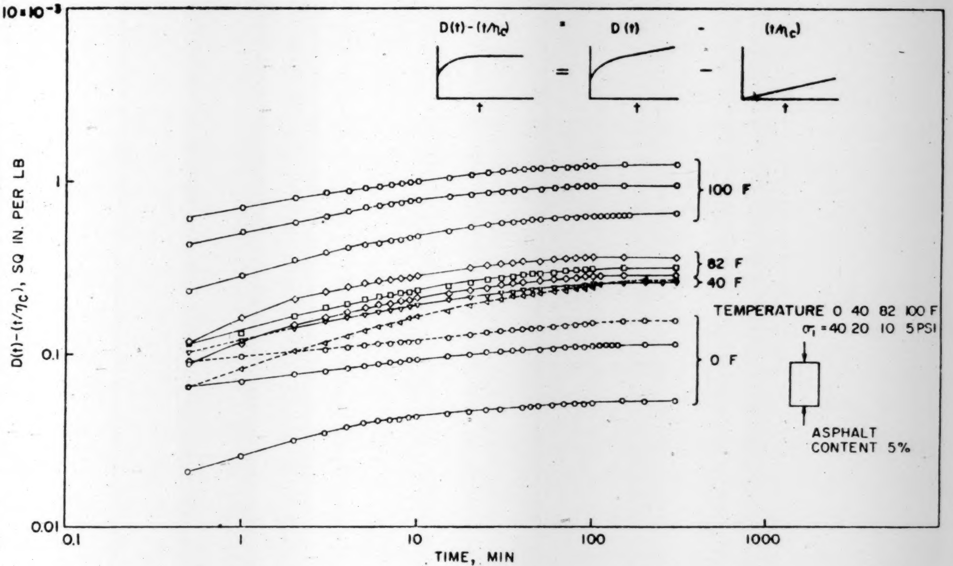


Fig. 5.—Creep Test—Log $[D(t) - (t/\eta_c)]$ Versus Log Time at 100 F, 82 F, 40 F, and 0 F for 5 Per Cent Asphalt.

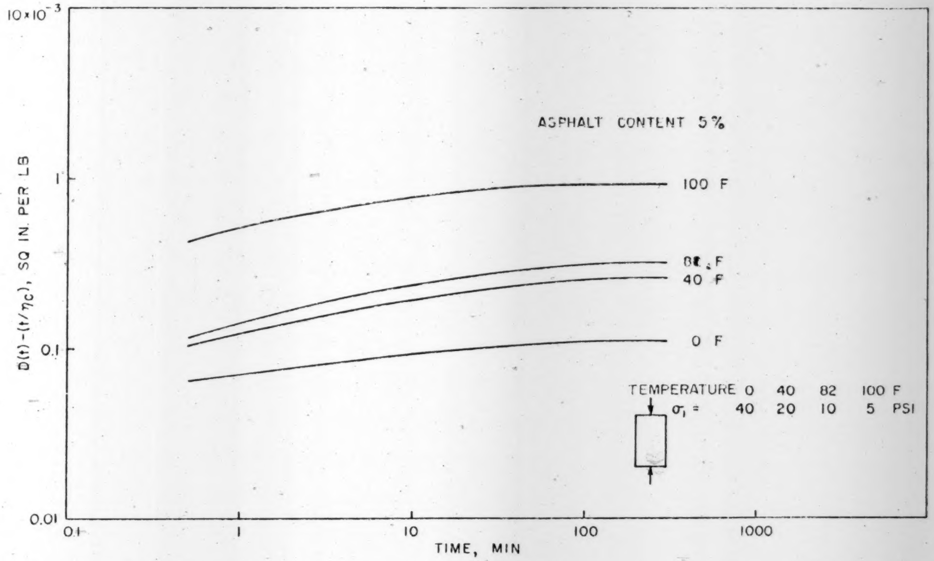


Fig. 6.—Creep Test—Average Curves of Log $[D(t) - (t/\eta_c)]$ Versus Log Time at 0 F, 40 F, 82 F, and 100 F for 5 Per Cent Asphalt.

tribution of aggregate size and aggregate type but different amounts of asphalt.

These curves all show a classical creep behavior which can be represented approximately by a four-element model. There was poor correspondence between creep and creep recovery, indicating that these mixtures deviate from linear viscoelastic behavior.

It was not possible to establish any

systematic difference in creep behavior for various asphalt contents since specimen variability produced effects greater than the differences observed for the different asphalt contents tested. The influence of asphalt content becomes evident at higher temperatures, but its effect is still relatively slight.

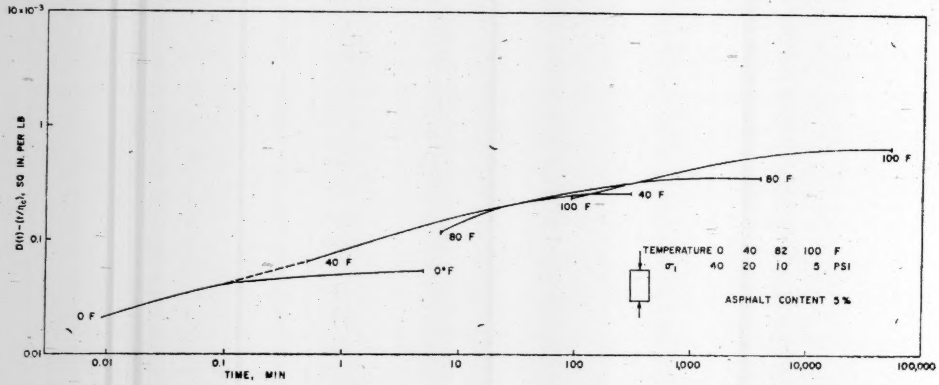


FIG. 7.—Creep Test—Typical Creep Curves After Horizontal Shift, Log $[D(t) - (t/\eta_c)]$ Versus Log Time for 5 Per Cent Asphalt.

As is evident from Fig. 4, if the experiment was allowed to continue for a long enough time, the low asphalt content mixtures exhibited apparent aggregate interlock, since deformation stopped completely. After a long time the particles are probably able to pierce the asphalt film and lock against one another. In the realm of linear viscoelastic theory there exists no adequate mathematical method for dealing with this phenomenon.

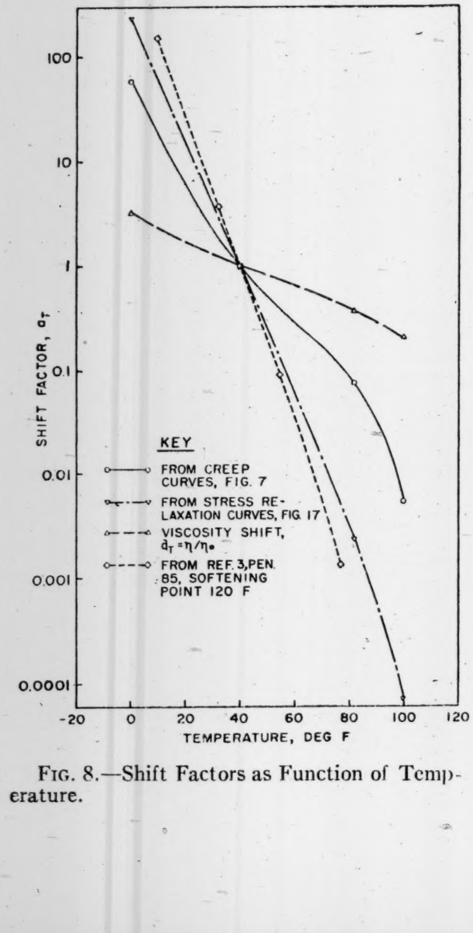


FIG. 8.—Shift Factors as Function of Temperature.

In the creep tests, difficulties were encountered in data reproducibility. (Fig. 5). In order to see the relationships between the curves at different temperatures more clearly a single average curve for each temperature was calculated by point-to-point averaging of the duplicate curves (Fig. 6). The stress applied at different temperatures was chosen to give approximately the same magnitude of final strain encountered at the highest temperature. In these and later plots of creep data, the steady-state flow component, t/η_c, was subtracted from $D(t)$.

Time-Temperature Superposition of Creep Curves.—In order to effect time-temperature superposition a horizontal shift of the curves in Fig. 6 has to be carried out. The reliability of the horizontal shifts can be judged by two factors: (1) the smoothness of the "master curve" obtained and (2) the magnitude of the a_T values as compared to the a_T values for pure asphalt.

It was possible to carry out an approximate horizontal shift by using selective curves in Figure 6 (Fig. 7). The master curve obtained in this way was not exactly smooth but the results indicate that this method of treating data can probably be applied in many cases.

The a_T values for this curve as a function of temperature are shown in Fig. 8. An attempt was made to correlate this curve with the a_T curves obtained for a large number of asphalts by Philippoff and co-workers at the Franklin Institute (see references (1,2)). Figure 8 shows the behavior of a_T versus temperature for a pure asphalt (2) from the study above which has characteristics similar to the asphalt used in the present experiments. These two a_T curves do not agree well, especially at higher temperatures.

Another method of determining a_T is by using the equation

$$a_T = \frac{\eta T_0 \rho_0}{\eta_0 T \rho}$$

where:

η_0 = viscosity of mix determined from the steady state flow region,

T_0 = reference temperature in deg K, and

ρ_0 = density at the reference temperature.

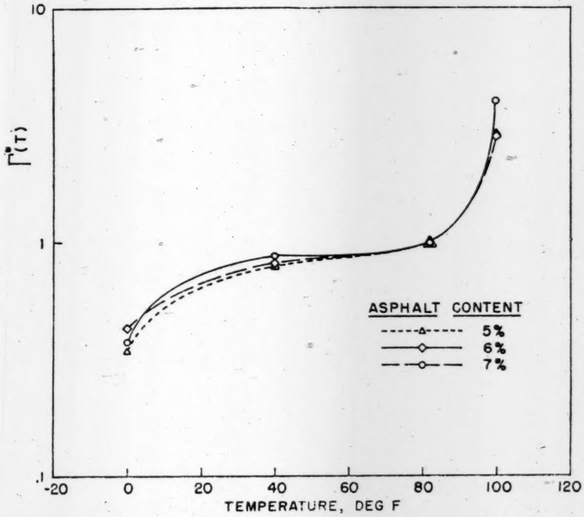


Fig. 9.—Creep Test—Vertical Shift Factor as Function of Temperature.

The values for a_T computed by this method, using the steady-state creep rates to define the viscosity, are in poor agreement with the a_T values for the pure asphalt. The computed curve is extremely flat and emphasizes the large deviation between the mix viscosity and the asphalt viscosity. See Fig. 8.

Vertical Shifts of the Creep Data.—The appearance of the creep curves in Fig. 6 suggests that a vertical shift, rather than a horizontal shift, might be appropriate.

The expression for the divergence from linearity for creep data can be written as

$$\begin{aligned}\epsilon(\sigma,t) &= \sigma \cdot D(\sigma,t)\\ &= \Gamma^*(\sigma)\cdot\sigma\cdot J(t) \quad \ldots\ldots (2)\end{aligned}$$

where:

$D(\sigma,t)$ = compliance in creep under axial stress,
$\Gamma^*(\sigma)$ = factor for the deviation from linear behavior in creep,
$J(t)$ = linear compliance function,
ϵ = strain,
σ = stress, and
t = time,

but it is also possible to write $\Gamma^*(\sigma)$ as $\Gamma^*(\sigma,T,t)$ because this deviational effect could be a function of time and temperature as well as of stress. In order to determine the form of the dependence of $\Gamma^*(\sigma,T,t)$ on time, temperature, and stress, it is necessary to hold two of the quantities constant and vary the third. Experiments of this type were conducted.

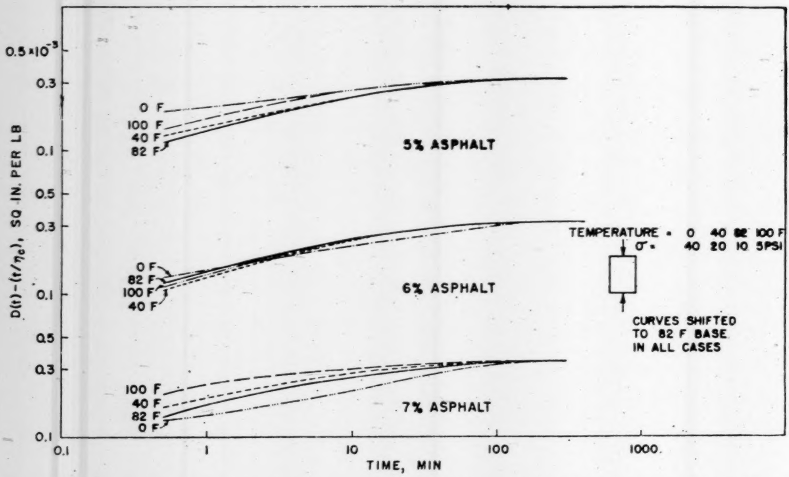


FIG. 10.—Creep Test—Vertically Shifted Log $[D(t) - (t/\eta_c)]$ Versus Log Time Curves.

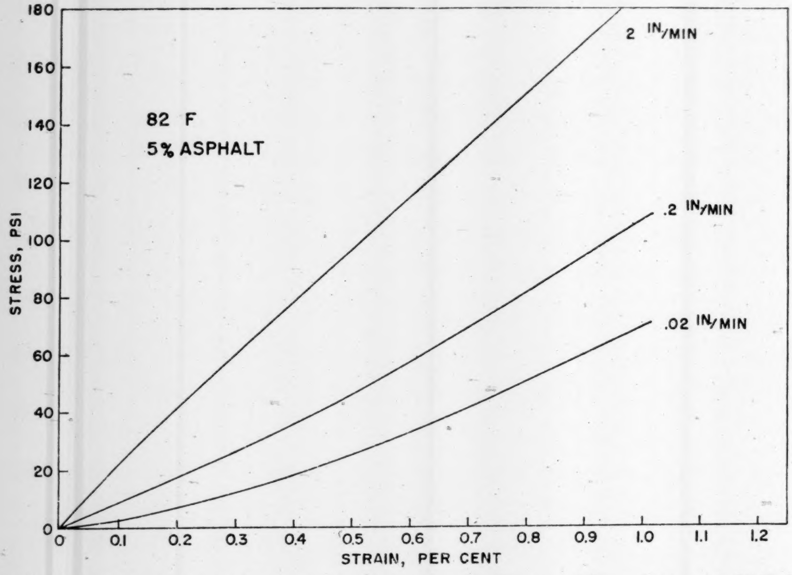
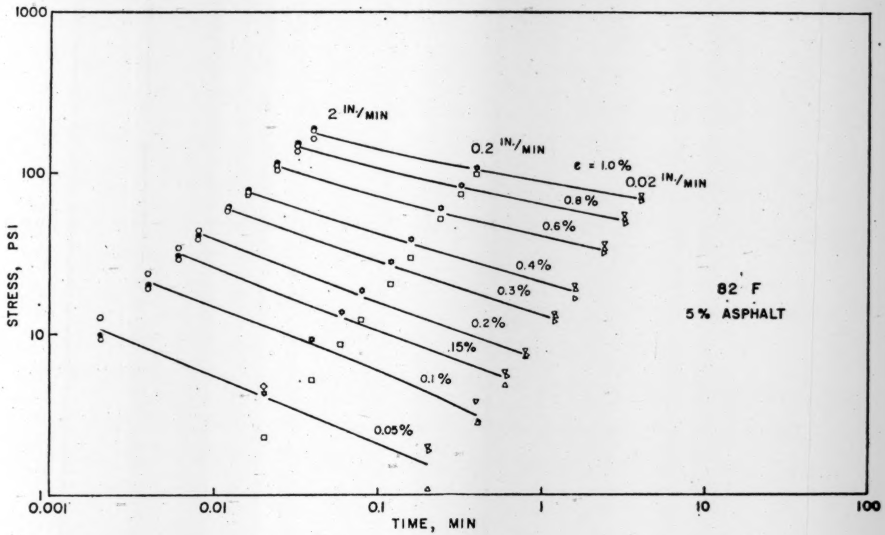
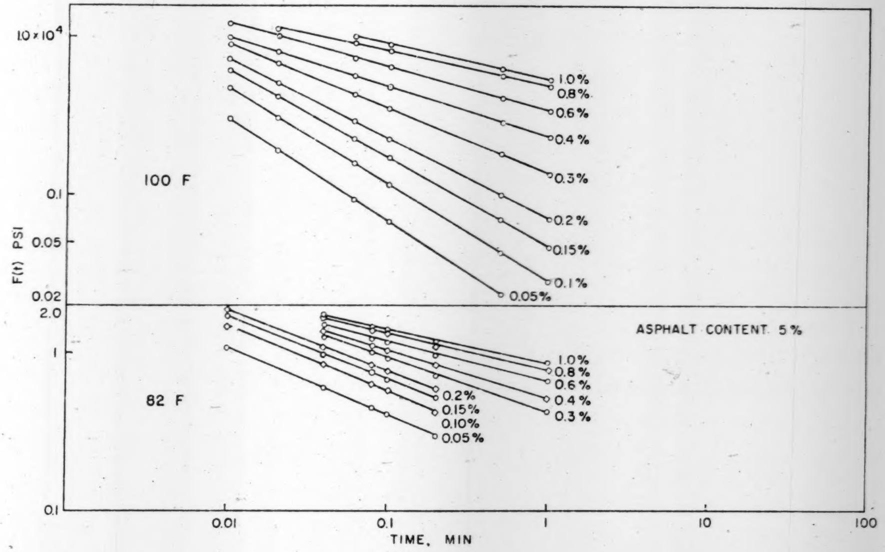


FIG. 11.—Constant Rate of Compression Stress-Strain Curves; Three Rates at 82 F for 5 Per Cent Asphalt.



(*a*) Check of applicability of Eq 3 at 82 F; 5 per cent asphalt.



(*b*) Typical $F(t)$ curves from constant rate of compression. Stress-strain tests at 82 F and 100 F, 5 per cent asphalt.

Fig. 12.—Constant Rate of Compression Test.

It was found that Γ^* was a function of temperature alone; therefore the deviation from linearity in creep is temperature-dependent only (Fig. 9). The fact that $\Gamma^*(T)$ is a function of temperature but is not a function of time is in itself a violation of basic viscoelastic principles. The implications of this will be discussed later.

In order to determine $\Gamma^*(T)$, the average creep curves were shifted vertically using 82 F as a reference base.

The shifted curves are shown in Fig. 10. There seems to be fairly good coincidence except for mixes with 7 per cent asphalt, where deviations begin to occur. The values of these shifts, which are $\Gamma^*(T)$, are shown in Fig. 9, which indicates the nature of the dependence of $\Gamma^*(T)$ on temperature for the three different asphalt contents.

Constant Rate of Compression:

Constant rate of compression tests have a distinct advantage in that the variables to be studied (stress, time, and temperature) can be more easily covered, in a fewer number of tests and in a considerably shorter period of time, than by stress relaxation tests. In constant rate of compression tests it is only necessary to run at three rates at any given temperature and the behavior is known for any value of strain from zero to the final strain. From the constant rate of compression tests it is possible to calculate the stress relaxation behavior, using viscoelasticity theory, provided the material is viscoelastic.

Stress-Strain Curves.—The first step in obtaining the stress relaxation curve from constant rate of compression tests is the running of stress-strain curves for at least three different rates (Fig. 11).

The effective modulus of the asphalt mixtures goes up with decreasing temperature and increasing rate of loading. The stress-strain curves are fairly straight lines in the temperature range 0 F to 82 F. See Figure 11. The closer together the stress-strain curves are at a given temperature, the less difference there is in the relaxation times that are being dissipated during the compression test. The amount of separation of the curves at various rates of loading is indicative of the amount of relaxation that is to be expected at the temperature in question.

Determination of Validity of Equation 3.—From the stress-strain curves of Fig. 12(*a*), it is possible to determine the validity of the following relation:

$$\begin{aligned}\sigma(\epsilon,t) &= \epsilon\cdot F(\epsilon,t)\\ &= \Gamma(\epsilon)\cdot\epsilon\cdot S(t)\ \ldots\ldots\ldots\ (3)\end{aligned}$$

$$\begin{aligned}\log\sigma(\epsilon,t) &= \log\epsilon + \log F(\epsilon,t)\\ &= \log[\Gamma(\epsilon)\cdot\epsilon] + \log S(t)\ldots (4)\end{aligned}$$

where $F(t)$ is the apparent constant rate of compression modulus, $\Gamma(\epsilon)$ is the factor for the deviation from linearity, and $S(t)$ is the linear modulus function under constant strain rate conditions. First a number of arbitrary strains are selected. At each strain value, a stress is obtained from the curve at each strain rate. The time required to reach the selected strain at each strain rate is given by $\epsilon/\dot{\epsilon}$. This combination of stress and time value is plotted in log-log form. If these iso-strain points all give parallel curves, then the data can be represented by Eq 4. Fig. 12(*a*) shows log σ versus log t curves at 82 F and 5 per cent asphalt content. The slope of the log σ versus log t curves gives an indication of the amount of relaxation in the system, and the vertical separation of these curves allows evaluation of $\log[\Gamma(\epsilon)\cdot\epsilon]$.

Tests were made at various asphalt contents but there were only slight differences between the 5 per cent and 7 per cent curves.

It is possible to make a separation of the time and strain effects according to Eq 4. Although this method will prove not to be totally valid, at least it will give some indication of the nature of the deviations from linear viscoelasticity and the cause of these deviations.

Nature of the Deviation from Linear Viscoelasticity (Stress Relaxation).—Just as it was possible in creep to write $\Gamma^*(\sigma)$ more generally as a function of stress, time, and temperature $\Gamma^*(\sigma,t,T)$, so it is possible to write $\Gamma(\epsilon)$ as $\Gamma(\epsilon,t,T)$. That is, the deviations from linearity can also be a function of other variables than strain alone. The dependence of $\Gamma(\epsilon,t,T)$

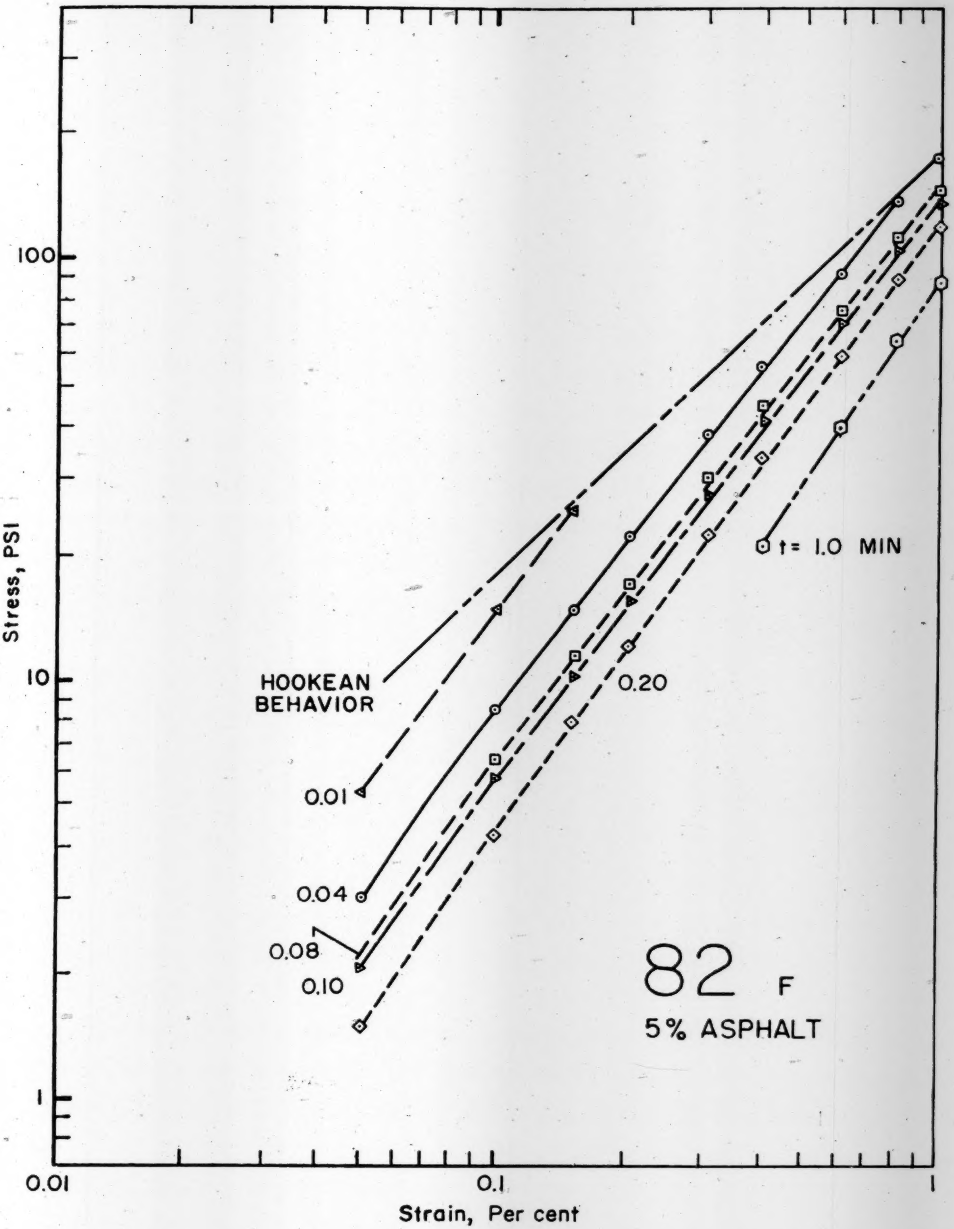


FIG. 13.—Constant Rate of Compression Test—Determination of Time-Dependence of $\Gamma(\epsilon, t)$; 82 F; 5 Per Cent Asphalt.

on these variables can be checked as before by holding two variables constant and changing the third.

corresponding values of stress and strain are plotted on log-log paper. By plotting log σ versus log ϵ, the shape of these particular curves represents $\Gamma(\epsilon)$. If these curves are all parallel when plotted as log σ versus log ϵ so that they can be shifted vertically along the stress axis, then $\Gamma(\epsilon)$ can be said to be independent

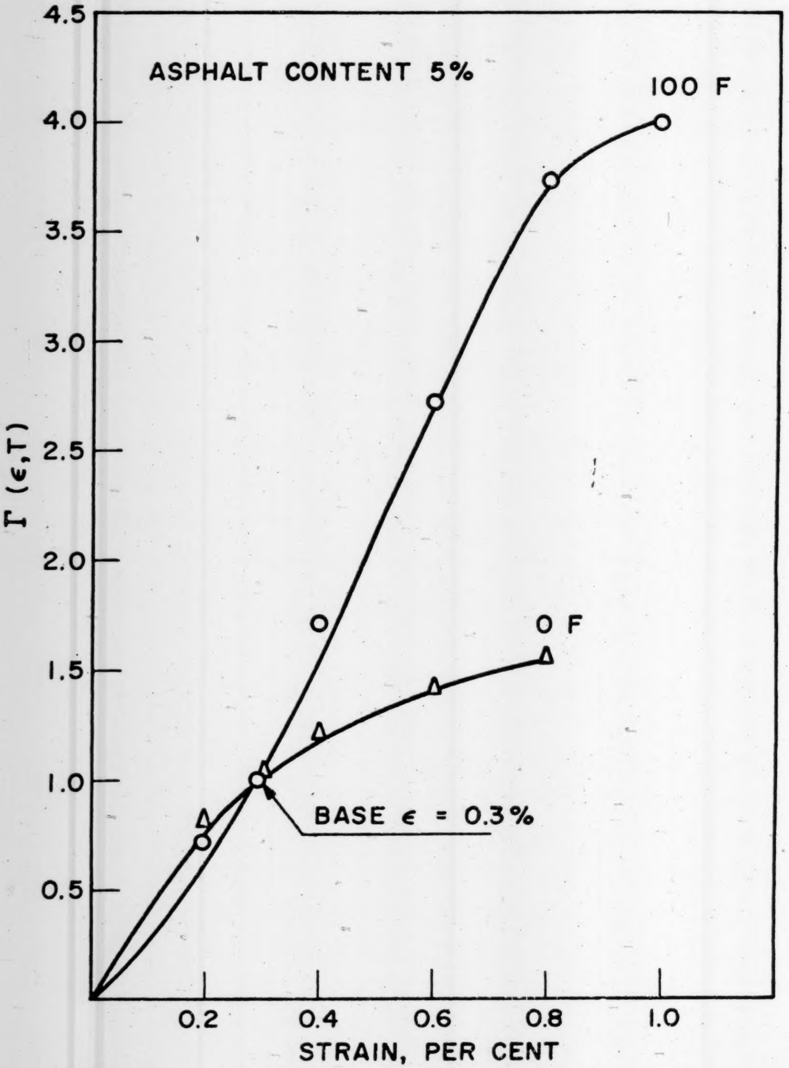


FIG. 14.—Constant Rate of Compression Test—Strain-Dependence of $\Gamma(\epsilon, t)$ at Two Temperatures; 5 Per Cent Asphalt.

$\Gamma(\epsilon,t,T)$ *as a Function of Time.*—It is possible to determine the time-dependence by taking isochronal values of stress and strain from Fig. 12(*a*). Arbitrary time values are chosen and the of time. Fig. 13 shows the isochronal plots for 82 F and 5 per cent asphalt.

At all temperatures, the curves start to be somewhat non-parallel at lower strains; this effect is only slight so that $\Gamma(\epsilon,t,T)$ can be considered to be independent of time, and therefore can be written as $\Gamma(\epsilon,T)$.

$\Gamma(\epsilon,T)$ as a Function of Temperature.—In order to determine whether $\Gamma(\epsilon,T)$ is a function of temperature, curves of log σ versus log ϵ were plotted for an arbitrary fixed time at various temperatures. These graphs are not shown. If $\Gamma(\epsilon,T)$ is not a function of temperature, the curves in such a plot should again be parallel (in the sense of a shift along the log σ scale). Three time values were chosen for examination: 0.01, 0.1, and 1.0 min. The 0.01-min curves show very

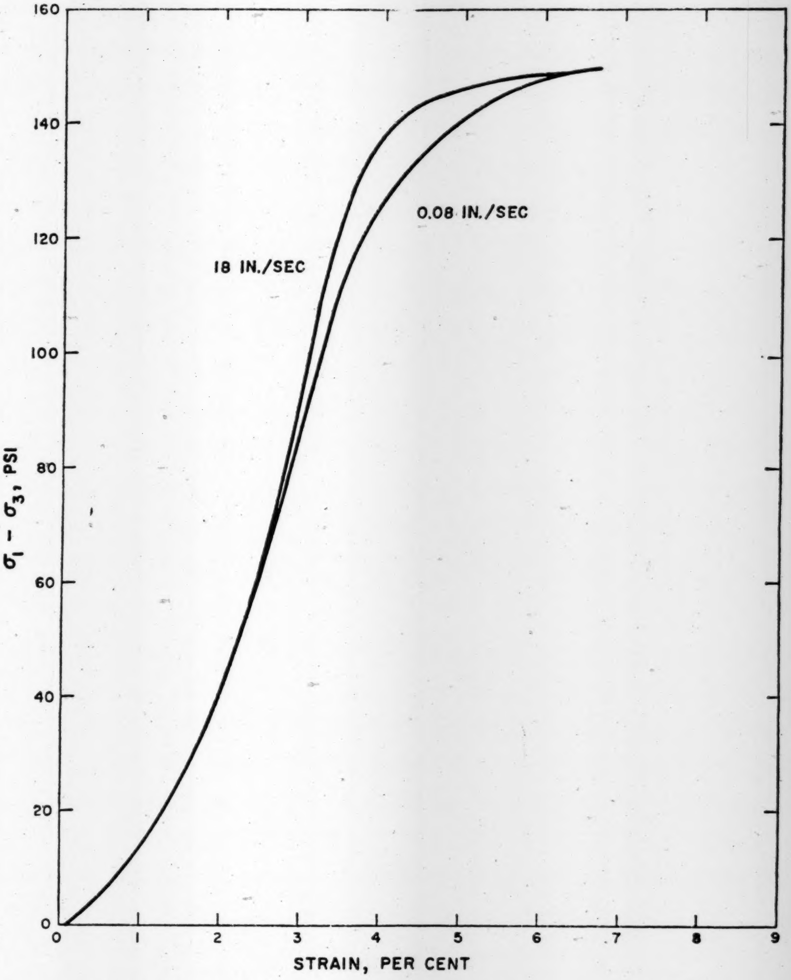


FIG. 15.—Stress-Strain Curves (Confined Compression), Plotted as $(\sigma_1 - \sigma_3)$ Versus ϵ, for Saturated Ottawa Sand at Two Rates of Compression (After Whitman and Healy (11)).

little temperature dependence of $\Gamma(\epsilon,T)$. This is not surprising as the isochronal time here is approaching that of a dynamic type of test. At high speeds viscous effects are decreased; that is, in terms of a mechanical model, if a dashpot is subjected to a very fast impulse, it tends to freeze up. Therefore there should be no time effects at sufficiently fast rates of loading. At longer times $\Gamma(\epsilon,T)$ seems to be more of a function of temperature, since the curves become less and less parallel. In the 1.0-min isochronal graph, not shown here, the 82 F and 40 F curves are reasonably parallel, indicating that $\Gamma(\epsilon,T)$ is not a strong function of temperature in this temperature interval. This is similar to the behavior noted in creep (see Fig. 9) where $\Gamma^*(T)$ was also not a strong function of temperature in this region. The $\Gamma(\epsilon,T)$ curves show a large temperature effect only at 100 F where the specimen begins to soften (see Fig. 14).

$\Gamma(\epsilon,T)$ *as a Function of Strain.*—We may consider finally whether $\Gamma(\epsilon,T)$ is a function of ϵ. It is possible to determine if it is strain-dependent from Fig. 12(*b*). In Fig. 12(*b*) at a given temperature the vertical separation of the iso-strain curves is $\Gamma(\epsilon)$, not $\Gamma(\epsilon,T)$, since T = constant. If there were no separation, then $\Gamma(\epsilon)$ would be unity, but it is fairly obvious from these curves that this is not the case.

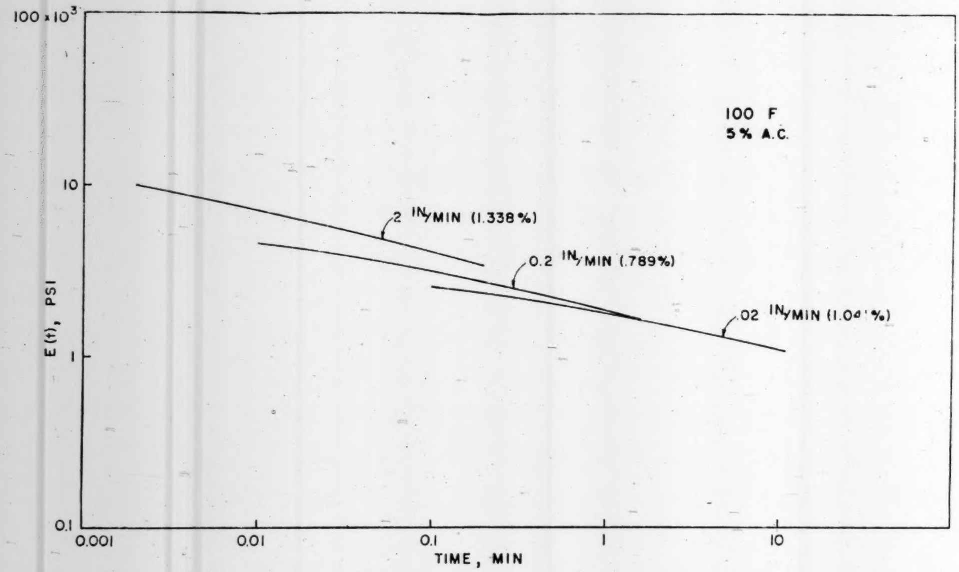


FIG. 16.—Stress Relaxation Test—Curves for Specimens Compressed to Fixed Strain at Different Rates; 5 Per Cent Asphalt at 100 F.

The values for $\Gamma(\epsilon)$ were obtained graphically by selecting a reference curve and shifting all the other curves vertically onto this curve. The shift values, $\Gamma(\epsilon)$, obtained in this way are shown in Fig. 14 for the 5 per cent asphalt mixtures. As temperature increases, $\Gamma(\epsilon)$ increases. At high temperatures, the asphalt approaches a liquid condition and $\Gamma(\epsilon)$ corresponds to some of the typical stress-strain behavior that is noted for water-saturated granular soils (Fig. 15).

Stress-Relaxation—Computed and Observed:

Directly Measured Relaxation Modulus E(t).—In the direct measurements of stress-relaxation, specimens were deformed in the Instron machine at a constant rate of compression up to the strain value desired for the relaxation test. Since a finite time is required to bring the specimen to its final fixed deformation, the deformation rate will affect the shape of the relaxation curve obtained, particularly in the short-time region. This is because relaxation can begin to take place during the initial deformation process. The slower the rate at which the specimen is brought to its final strain level, the less relaxation the $E(t)$ curve will show initially. If linear viscoelasticity holds, relaxation curves at different strains and different strain rates should all coincide at longer times, when plotted as $E(t)$.

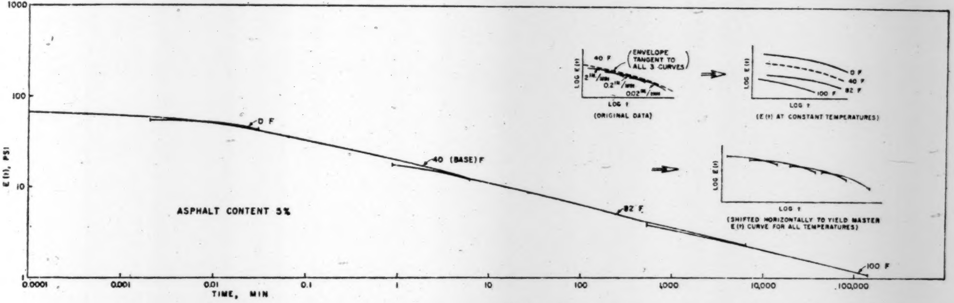


FIG. 17.—Superposition of Relaxation Curves at Different Temperatures by Horizontal Shifting to Give Master Relaxation Curve; 5 Per Cent Asphalt.

The curves in Fig. 16, although run at slightly different strains, coincide at long times and do not show the nonlinear vertical separation expected from the behavior observed in the constant rate of compression tests. In addition, the scatter observed in the stress-relaxation test was considerably less than in any of the other types of test used in this investigation. Since linear behavior is observed after long elapsed times, this appears to indicate that the observed viscoelastic behavior may be entirely that of the asphalt component. In a constant strain test of this sort, it probably will not be necessary for the aggregate particles to move relative to one another. If these experimental relaxation tests only involve the relaxation of the asphalt, we should be able to shift these curves horizontally and determine an a_T curve using methods commonly applied to viscoelastic polymeric materials.

In order to use relaxation curves at different strain rates, it was necessary to draw an envelope to the curves to correct for the short-time relaxation due to the finite loading rates. The curves then could be shifted horizontally and the shift factors a_T calculated in the usual way. The envelope curves at constant temperature are shown in the upper part of Fig. 17 before shifting.

The composite or master curve obtained by shifting the curves is also shown in Fig. 17. Good superposition of the component curves was obtained. The shift factors determined from the master curve in Fig. 17 were plotted versus temperature together with a_T values for pure asphalt that were determined at the Franklin Institute (see Fig. 8). The agreement between the two sets of a_T values is very good throughout the whole temperature range. This offers support for the hypothesis that the relaxation behavior of this type of composite is due almost entirely to the viscoelastic process in the asphalt phase. In direct stress-relaxation, the relaxation is not a function of strain. This and the good agreement with a_T values for pure asphalt would indicate that the $\Gamma(\epsilon)$ function is unity for this type of test.

Comparison of E(t) Values (Direct Versus Computed).—First it was necessary to calculate $E(t)$ from the $F(t)$ values so that a comparison could be made with the directly measured stress-relaxation curves. This was done by the relationship derived for linear viscoelasticity by Smith (**10**):

$$E(t) = F(t)\left[1 + \frac{d \log F(t)}{d \log t}\right] \ldots (5)$$

Since the deformation in the relaxation tests was not applied instantaneously, as previously discussed, the initial portion of the relaxation spectrum has some uncertainty associated with it. However, it would be expected that the computed and the observed relaxation curves should eventually converge if the procedure of computing $E(t)$ from $F(t)$ is correct. The curves do not seem to converge. The computed curves always lie above the direct experimental curves, as shown in Fig. 18. It seems that these two tests are not interconvertible. This is due to the fact that in direct relaxa-

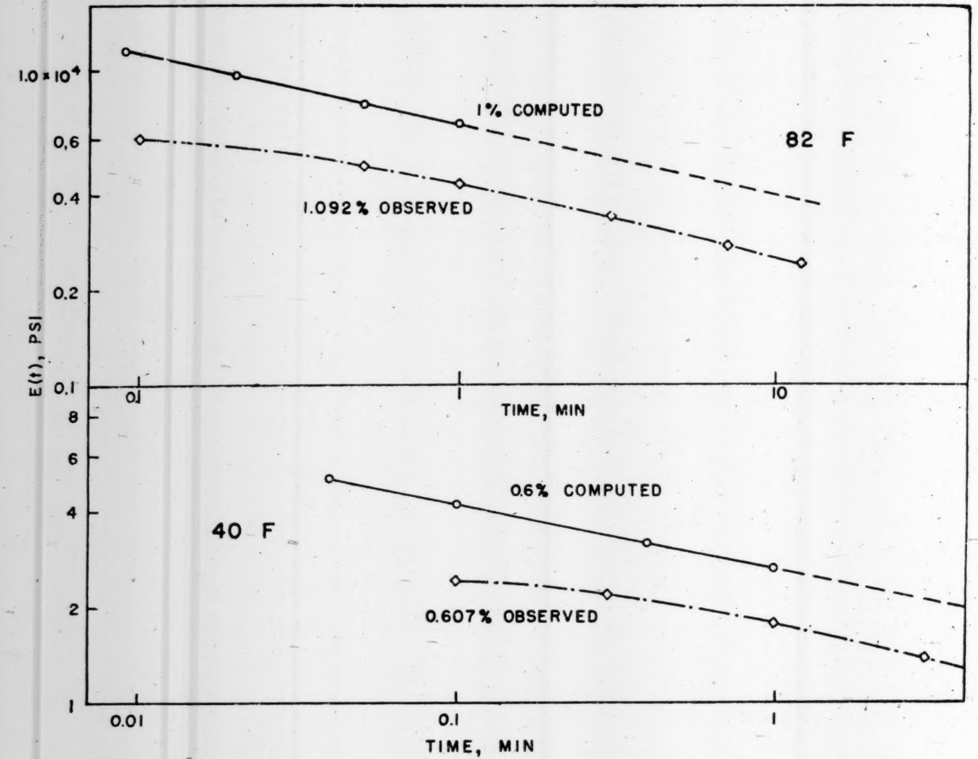


FIG. 18.—Comparison of Stress Relaxation Curves Computed from Constant Rate of Compression Data with Directly Observed Relaxation Curves; 40 F and 82 F; 5 Per Cent Asphalt.

tion tests there is no need for relative particle movement to permit relaxation. However, in the constant rate of compression tests the aggregate is forced to move, producing interparticle friction and associated stresses which contribute to the total observed stress in this type of test. The interparticle friction, therefore, contributed to $F(t)$, and the $E(t)$ values computed from $F(t)$ will have a component from this effect. This would explain why the computed values of $E(t)$ are higher. For filled systems it does not seem likely even from an intuitive consideration of the physical mechanisms involved that the constant rate of compression and the direct relaxation tests are comparable.

DISCUSSION

Disturbance of the Viscoelastic Properties of the Asphalt by Aggregate:

The introduction of the large volume (80 per cent) of solid filler (aggregate) into asphalt, ordinarily a linear viscoelastic material, produces a material with nonlinear and pseudoviscoelastic properties. Although the asphalt-aggregate composition retains some of the properties of the viscoelastic cementing agent, the difference between the viscoelastic response of the asphalt alone and that of the composite seems to be a strong function of the type of test employed. The more aggregate displacement involved, the greater is the difference between the viscoelastic properties of the composite and of the asphalt.

The first indication of the aggregate's effect in changing the characteristics of the asphalt is observed in the creep tests where aggregate interlock takes place and the deformation ceases. Certainly "pure" asphalt would never show such an effect.

The large differences between the instantaneous deformation and instantaneous recovery can also be directly attributed to the effect of the aggregate. During the creep deformation, the asphalt film is forced out from between the particles. Upon removal of the load there is less asphalt film present to accomplish an elastic recovery equal to the instantaneous deformation.

Wada and Hirose found that for asphalts the shift factor, a_T, could be described by the ratio of the asphalt viscosities at different temperatures. However, when this same procedure was tried for the viscosity of the asphalt-aggregate composites in the present investigation, the computed a_T values differed widely from the a_T values for the asphalt. The interparticle contacts of the aggregate are apparently decreasing the temperature dependence of the viscosity of the mix to such a pronounced degree that the above relation is no longer valid.

The action of the aggregate could also be used to explain the difference between the $E(t)$ computed on the basis of the constant rate of compression test and the directly observed $E(t)$. In the former case the motion of the aggregate was forced, which resulted in higher stress, but in the latter case it was possible for the relaxation to occur without the need of aggregate motion.

Pseudo and Real Viscoelastic Effects Noted in the Composite:

Since the asphalt occupies 12 to 17 per cent of the mix by volume, the composite might be expected to exhibit some form of viscoelastic behavior, but most of the behavior observed seems to be pseudoviscoelastic. This pseudoviscoelastic behavior is illustrated by the creep data. It appears that the creep curve could be characterized by a generalized Voigt model; however, the fact that very little recovery is observed invalidates the representation of the creep behavior of the composite by this model.

The ability to make a horizontal shift of the creep data also would seem to indicate ideal viscoelastic behavior. However, the lack of smoothness and complete superpositon in the master curve obtained, and the poor agreement of the a_T obtained from creep data with those for pure asphalt, again indicate that this is pseudoviscoelastic behavior.

There are indications, however, that the composite material can exhibit true viscoelastic behavior in a direct stress-relaxation test. The superposition of curves from this type of test is considerably better than in the case of the creep tests. The resulting master curve for stress-relaxation is quite smooth, and the a_T values agree with those for pure asphalt. In this direct relaxation test, where the aggregate is probably not required to move, the relaxation can be described in terms of the properties of the asphalt to a very large extent.

Nature of the Deviations from Linear Viscoelasticity:

From the total (composite) relaxation and creep functions it was possible to separate out two Γ functions that did not depend on time. These non-time-dependent Γ functions represent the nonlinear effects. The nature of these functions, in regard to their dependence on stress, strain, time, and temperature, has been established here. Since the asphalt is a linear viscoelastic material, the nonlinear effects encountered in the asphalt-aggregate compositions are attributed to the aggregate. The Γ function which characterizes the nonlinear effects should then be related to the stress-strain behavior of the granular network. The fact that the Γ functions for creep and constant rate of compression are time-independent but are temperature-dependent immediately suggests that these functions are nonviscoelastic in nature. Linear viscoelasticity requires that time and temperature be interrelated; thus all viscoelastic phenomena that are time-dependent will also be temperature-dependent.

It has been established that the stress-strain functions for densely packed granular sands are independent of time (see reference 11). If the Γ functions for the asphalt-aggregate are related to the stress-strain behavior of dense packed granular soils, then the Γ functions should in fact be independent of time. The reason why the stress-strain function for a granular soil is not time-dependent is that in a densely packed sand of almost uniform particle size, no matter what the deformation rate is, the particles cannot move out of the way of each other. In order for the particles to follow a very rapid deformation they would have to climb or jump over one another.

In asphalt mixtures at higher temperatures the asphalt viscosity decreases and the nonuniformly sized aggregate particles are able to move more easily past one another. As the temperature decreases two effects occur: (1) the viscosity of the asphalt increases which hinders the aggregate particles from slipping by each other, and (2) the aggregate particles are forced to move in large aggregate combinations which now approximate the uniform sand particle behavior. Therefore, the details of the aggregate motion depend on the temperature, and as a consequence the Γ functions are temperature-dependent. At higher temperatures where the viscosity of the asphalt is quite low, the Γ function approaches the form of the stress-strain function characteristic of soils, indicating that interparticle interactions are providing the main contribution to the observed stress in this region.

It has been noted that the Γ function in the constant rate of compression test is a function of strain, while the Γ function in the creep test is not a function of stress. The reason is that the two types of test are not comparable. In the constant rate of compression test the deformation is continuously forced, which involves displacing particles that offer resistance, and as a result there is a strain-dependence. In the creep test, although the initial deformation is forced (the initial elastic component), the deformation which occurs is not completely forced but will seek the path of least resistance. There exists in the creep test the possibility of a total lock-up occurring in the aggregate network (this did occur for 5 per cent asphalt content at high temperatures) but a constant rate of compression test would not allow this type of total locking to occur since the continuous deformation forcibly breaks up these lock-ups (probably even of a localized type). Therefore, the Γ function for creep does not have to be a function of stress as much as the Γ function for the constant rate of compression has to be a function of strain. We may assume that the Γ functions always represent interparticle effects, or effects associated in some way with the presence of the particles.

Influence of the Type of Test on the Rheological Properties:

The rheological properties of a material may be characterized by the response of the material to various types of imposed forces or deformations. In dealing with ideal viscoelastic materials these properties are all interrelated, but in composite systems this interrelation does not appear to hold.

The Γ functions which represent deviations from ideal linear viscoelastic behavior play a more important role as the behavior of the aggregate is more involved in the deformation. The more the aggregate is involved, the greater the dependence of Γ on various factors such as stress or strain, time, and temperature. In the constant rate of compression test, where the aggregate is moved forcibly, Γ is a function of strain and temperature; in the creep test, where the aggregate motion is less forced, Γ is a function only of temperature; but in the stress-relaxation test, where there seems to be no aggregate motion required, there are indications that the Γ function does not play a role at all.

CONCLUSIONS

1. Asphalt-aggregate composites can show different types of rheological behavior, depending on the type of experiment involved. Although pure asphalt is a linear viscoelastic material, the composites generally show nonlinear viscoelastic behavior, and even behavior which is not viscoelastic.

2. The departures from viscoelastic behavior appear to be associated with interparticle contacts of the aggregate. These contribute to the stress required to cause deformation of the system, and in extreme cases can lead to a "locking" (for example, in creep experiments at constant applied stress) such that no further deformation of the system takes place. While the interparticle effects appear to be negligible in stress relaxation at constant strain, they are observed in creep experiments and are seen even more strongly in measurements of compression stress-strain curves. As a consequence, these three types of data are not interconvertible in the way that linear viscoelastic data would be. The temperature-dependence of the three types of data is also different, because the interparticle contribution to the stress is less temperature-dependent than the viscoelastic contribution.

3. Although the nonlinear viscoelastic behavior can be handled, in general, by theoretical formulations already available, there is no satisfactory theory at

present which would allow an adequate description of the interparticle effect. The type of theory needed for the latter would probably be very similar to the type of theory needed to describe the rheological behavior of water-filled granular soils.

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APPENDIX

PHYSICAL PROPERTIES OF THE MATERIALS INVESTIGATED

Mix Design:

The asphalt-aggregate mixture used conformed to Massachusetts Type I specifications for top-course composition. The various gradations and mix proportions are given below:

BASIC MIX, 6 PER CENT ASPHALT MIX.

Through	Retained	Per Cent	Weight of Fraction, g	Total Accumulated Weight, g
½ in.	⅜ in.	5	180	180
⅜ in.	No. 4(⅜ in.)	15	540	720
⅜ in.	No. 4(4)	15	540	1260
No. 4	No. 10	18	647	1907
No. 10	No. 20	9	324	2231
No. 20	No. 40	10	360	2591
No. 40	No. 80	12	433	3024
No. 80	No. 200	4	144	3168
No. 200 to Pan (Filler)		6	216	3384
Asphalt	...	6	216	3600

	7 Per Cent Asphalt Mix	5 Per Cent Asphalt Mix
Weight of asphalt,[a] g	255	178
Total accumulated weight of mix, g	3639	3562

[a] Weight of aggregate remains the same for all mixes.

Type of Components and Their Specific Gravities:

The coarse aggregate was clean hard crushed stone (basalt) from the Massachusetts Broken Stone Quarry in Weston, Mass. The sand was also obtained from this quarry which maintains a pit in Acton, Mass. The filler material was stone dust derived from the same coarse aggregate. The specific gravities of the various components were:

	Specific Gravities	ASTM Methods of Test
Coarse aggregate (crushed stone)	2.92	C 127 – 59[a]
Fine aggregate (sand)	2.53	C 128 – 59[b]
Mineral filler (stone dust)	2.94	C 188 – 44[c]
Asphalt	1.008	D 70 – 52[d]

[a] Method of Test for Specific Gravity and Absorption of Coarse Aggregate, 1961 Book of ASTM Standards, Part 4.

[b] Method of Test for Specific Gravity and Absorption of Fine Aggregate, 1961 Book of ASTM Standards, Part 4.

[c] Method of Test for Specific Gravity of Hydraulic Cement, 1961 Book of ASTM Standards, Part 4.

[d] Method of Test for Specific Gravity of Road Oils, Road Tars, Asphalt Cements, and Soft Tar Pitches, 1961 Book of ASTM Standards, Part 4.

The asphalt was obtained from the Esso Refinery, Everett, Mass., which uses a Venezuelan crude oil. All the asphalt for these tests was obtained at the same time from the same distillation run to insure uniformity. The properties of the asphalt were:

Penetration[a]	82 to 85
Softening point, deg F[b]	118 to 120
Viscosity, poises:	
at 0 F	2.8×10^{10}
at 40 F	1.1×10^{8}
at 82 F	1.2×10^{6}
at 100 F	2.7×10^{5}

[a] Method of Test for Penetration of Bituminous Materials (D 5 – 61), 1961 Book of ASTM Standards, Part 4.

[b] Method of Test for Softening Point by Ring and Ball Apparatus (E 28 – 58 T), 1961 Book of ASTM Standards, Parts 4, 7, 8, and 11.

Densities of the Compacted Mix:

The densities of the compacted mix are a function of the asphalt content when the same compactive effort is used in making the specimens. The specific gravities for the various asphalt mixes were:

Asphalt Content, per cent	Specific Gravity	ASTM Test Method
5........	2.36 to 2.38	D 188 - 53[a]
6........	2.39 to 2.42	D 188 - 53[a]
7........	2.43 to 2.45	D 188 - 53[a]

[a] Discontinued; replaced by Methods D 664 and D 974, 1961 Book of ASTM Standards, Part 7.

The above density ranges are the variations encountered in 25 samples at each asphalt content. The values of the densities were determined by water immersion.

Void Contents:

The void contents were computed on the basis of solid volumes.[4] The void contents were also a function of the amount of asphalt used and are listed below:

Asphalt Content, per cent	Per Cent Voids
5.............	6.3 to 7.1
6.............	3.2 to 4.4
7.............	0.8 to 1.6

The above void ranges are the limits of the variations encountered in 25 samples.

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[4] "Mix Design Methods for Hot Mix Asphalt Paving," *Asphalt Inst. Manual Series, No. 2*, p. 162.

EVALUATION OF LABORATORY TECHNIQUES OF SOAKING SOIL-ASPHALT SPECIMENS*

BY MORELAND HERRIN,[1] PHILLIP G. MANKE,[2] AND ZACHARIAH GEORGE[3]

There is at present no one universal standard for designing bituminous-stabilized soils. Not only are there a number of methods of design available, but also there are many different procedures and types of apparatus for evaluating the strength of the mixture. Usually the strength test is conducted upon specimens that are in a condition as bad or worse than the conditions expected in the field. Since high moisture content can be present under actual conditions and thus cause a reduction in strength of the soil-bituminous mixtures, various methods of soaking the laboratory compacted specimens have been devised in order to represent field conditions in the laboratory testing procedures.

Fundamentally, the theory dealing with stresses in any material is based upon the assumptions that the material is isotropic and homogeneous. It is essential, then, that in the evaluation of soil-bituminous mixtures, the mixture be the same throughout the specimen; this includes having the same moisture content throughout the specimen. Any variation in moisture content in a specimen between the inside and the outside or between the top and the bottom may result in misleading evaluation of the strength properties of the material. This, in turn, could lead to an erroneous design of the material and to an underdesigned pavement.

This study, then, was an attempt to evaluate some of the more common methods of soaking soil-asphalt specimens. Its primary purpose was to determine how thorough the different methods were for soaking soil-asphalt specimens. In addition, it was desired to study the influence of time and other factors upon the soaking process. In the future, after subsequent related studies, it may be possible that a method of soaking soil-asphalt specimens can be standardized that will be less time-consuming and more thorough than the present methods.

METHODS OF SOAKING SOIL-ASPHALT SPECIMENS

Of the various soaking methods in use, there appear to be three basic methods which have been used most prevalently—the total immersion method, the one-half immersion method, and a method in which the specimens are subjected to a vacuum-saturation process.

Perhaps the most common method of soaking soil-asphalt specimens is the total or complete immersion method, in which the specimens are completely submerged in water at room temperature to a depth of 1 in. above the top of the specimen and soaked for various periods of time. This method is specified in the

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ASTM procedure for designing soil-bituminous mixtures.[4] Both Rhodes (1)[5] and Anderton (2) proposed the total immersion method of soaking in their work with tar-soil mixtures. Rhodes used an 18-hr soaking time, while Anderton let his specimens soak for 14 days. The ASTM procedure as well as many highway departments, however, specifies a 7-day soaking period.

In the one-half immersion method used by Roediger and Klinger (3) and later by Benson and Becker (4) in their research on soil stabilization, the specimens are immersed in water to one-half their height and allowed to soak in a controlled-humidity room. Roediger and Klinger soaked their specimens for 7 days, while Benson and Becker used a 5-day soaking period.

TABLE 1.—SIEVE ANALYSIS OF SOIL MIXTURE.

Sieve Size	Per Cent Passing
No. 10	100
No. 40	96.7
No. 80	64.1
No. 200	37.3

TABLE 2.—PROPERTIES OF THE SOIL.

Atterberg limits:	
Liquid limit, per cent	21
Plasticity index, per cent	5
Fluff point range:	
Lower, per cent	5.5
Upper, per cent	13.0
Maximum density (standard Proctor), lb per cu ft	125.5
Optimum moisture content, per cent	11.0

The vacuum-saturation method, as it is referred to in this paper, was used by Holmes and Klinger (5) and also by Thurston (6) in his method for testing the stability of soil and liquid asphalt mixtures. In this method the specimens are placed in a vacuum chamber for 10 min and then submerged in water for a period of 24 hr.

A few other methods of soaking specimens are mentioned in the literature. The "capillary absorption test," used by McKesson (7), Hovis (8), and Anderton (2), allows the specimen to absorb moisture like a wick. Also, a variation of the one-half immersion method was used by Heriot (9) in which the specimens were immersed to a depth of one-fourth their height.

For the purposes of this study, it was deemed advisable to investigate only the first three methods.

MATERIALS AND LABORATORY PROCEDURE FOR PREPARING SPECIMENS

Soil:

The soil, an AASHO A-4 type, was obtained from a roadside bank near Stillwater, Okla. It is typical of the soils stabilized with asphalt in the state of Oklahoma and meets Oklahoma State Highway Specifications, Section 313, for Soil-Asphalt Base.[6]

Table 1 shows the results of the wet sieve analysis of this soil and Table 2 lists the related soil properties.

Asphalt:

The asphalt was a medium-curing liquid asphalt of ASTM grade MC-3[7] containing 14.5 per cent hydrocarbon volatiles by weight.

Normally the soil was stored in the laboratory and thus was in an air-dry condition when used. At least 24 hr before the soil-asphalt mixture was to be made, moisture was added to the soil to increase its moisture content to 8 per cent, which is within the fluff point range. At this moisture content, the soil had optimum mixing characteristics. The moist soil was put through a No. 10 sieve to break up the large particles, sealed in air-tight containers, and stored for at least 24 hr while the moisture dispersed throughout the soil mass. Although there were slight moisture losses during some of the processing, the moisture content was not a variable in this test.

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[4] Method of Testing Soil-Bituminous Mixtures (D 915), 1961 Book of ASTM Standards, Part 4.

[5] The boldface numbers in parentheses refer to the list of references appended to this paper.

[6] Standard Specifications for Highway Construction, Oklahoma State Highway Commission, 1958, p. 127.

[7] Specification for Cut-Back Asphalt (Medium Curing Type) (D 598), 1961 Book of ASTM Standards, Part 4.

The moist soil and $5\frac{1}{2}$ per cent (dry soil weight basis) of the asphalt cutback were mixed for a period of 2 min in a Hobart C-100 mixer. After passing the mixture of soil and asphalt through a No. 4 sieve to break up any large lumps, it was dried in an electric oven at 110 F for various periods. Immediately upon removal from the oven, the mixture was compacted into specimens.

In order that the effect of shape of specimen might be evaluated, specimens of two different shapes were compacted. Some specimens had diameters larger than their heights (that is, Hveem stabilometer specimens), while the other specimens had heights greater than their diameters. These specimens will be referred to as A and B, respectively, and were compacted as follows:

Shape A:

The Shape A specimens were standard Hveem specimens and were compacted by the Hveem gyratory method (**10**). These specimens were 4 in. in diameter and approximately 2 in. in height.

Shape B:

The rational size specimens were 1.86 in. in diameter and 4.5 in. in height. These specimens were formed by a double-plunger compaction method. A constant pressure of 3032 psi was applied in a universal hydraulic testing machine for 1 min to form these specimens.

After molding, each specimen was numbered and its weight and average height recorded. They were then immediately put to soak in the desired manner.

METHODS OF SOAKING SAMPLES

In this investigation three methods of soaking were evaluated: total immersion, in which the specimens were completely immersed; one-half immersion, in which the specimens were immersed one-half their height in water; and vacuum-saturation, in which the specimens were first subjected to a vacuum and then allowed to soak while completely immersed in water.

Total Immersion Method:

For the total immersion method the specimen rested on a special aluminum tray with closely spaced perforations and mounted on leveling screws.

After the tray was set in a shallow pan and leveled, six compacted specimens were placed on the tray and covered with water. The water level was adjusted and maintained at a height of 1 in. above the top of the specimens for the entire test. Two specimens were allowed to soak at room temperature for 2 days, two for 4 days, and two for 7 days. At the end of each soaking period, two specimens were removed from the pan and the free moisture removed from the surfaces by blotting. The specimens were then weighed and the average height of each determined.

Immediately, samples of the soaked material were obtained at appropriate locations within the specimens for determination of moisture content of the inner and outer portions of the sample.

For Shape A specimens, a core was cut from the specimens by means of a 2-in. inside-diameter cylinder with a sharpened edge which was pressed through the center of the specimens. The core, thus removed, measured approximately 2 in. in diameter and 2 in. in height. These cores were laid on their sides and cut into thirds with a knife. One-hundred-gram samples were taken for moisture determinations from the inner third of the core and from the combined two outer two thirds. Shape B specimens were cut vertically so that an inner core remained that was about 1 in. in diameter, or one-half the diameter of the original specimens. Moisture samples were taken of the material that had been cut in the outer portion. Another sample was taken from the center portion of the inner core. These samples were processed immediately for determination of their moisture content. The procedure for determining the moisture content is given in the Appendix.[8]

One-Half Immersion Method:

The one-half immersion method of soaking was carried on in a moist room. The specimens were placed on the aluminum tray and the height in the pan adjusted so that the specimens were one half in the water and one half in the humid air. As in the total immersion method, two specimens were allowed to soak for 2 days, two for 4 days, and two for 7 days. At the end of each soaking period, 2 specimens were removed, the surface moisture blotted, and the height and weight recorded.

A 2-in. diameter core was again removed from the center of each Shape A specimen. Each core was cut into thirds. Unlike the total immersion method, however, the moisture determinations were made on the samples taken from the top, middle, and bottom thirds of the core. Moisture samples were taken from Shape B specimens by laying each specimen flat on the table and cutting it into three equal lengths. The outer portion of the bottom and top thirds was each used for different moisture samples. A third sample was obtained from the inner core of the middle section that had been carefully separated from the outer portion by a knife. One hundred grams of each of these samples was crumbled and the moisture content immediately determined by the procedure outlined in the Appendix.

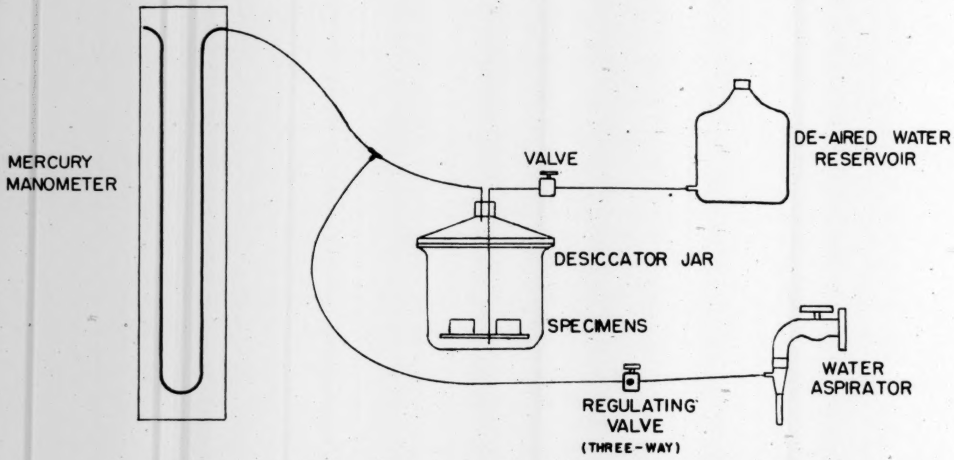

FIG. 1.—Schematic Diagram of Apparatus for Vacuum-Saturation Method.

Vacuum-Saturation Method:

The vacuum-saturation method of soaking used in this investigation was similar to the method proposed by Holmes and Klinger (5). However, preliminary investigation indicated that their method was too harsh for the soil-asphalt mixture being used, since specimens soaked in their manner were so soggy that they crumbled upon being removed from the water. Consequently, an adaptation of this method was used in order to obtain less critical conditions and to allow the study of the influence of several variables upon this soaking process.

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[8] See p. 1302.

After molding, specimens were placed in a desiccator jar that was used as the "water absorption box." This desiccator jar was connected to three lines, one attached to an aspirator for producing a vacuum, another to a manometer, and the third to a jar containing de-aired water (Fig. 1). With the valve in the line to the de-aired water closed, a vacuum was applied to the samples in the desiccator by means of the aspirator. The absolute pressure in the desiccator was measured by the manometer. After the desired period of applying the vacuum, the de-aired water was admitted to the jar and was allowed to cover the specimens. During this time, the vacuum was held constant within the desiccator. Immediately after the specimens were covered with the water, the jar was brought to atmospheric pressure and the specimens were allowed to soak for various periods of time.

TABLE 3.—MOISTURE CONTENT, TOTAL IMMERSION METHOD, PER CENT.

Specimen	Part of Specimen Tested	Days of Soaking			
		0	2	4	7
	0 hr in Drying Oven				
A...	Outside ⅔ of core	7.08	7.65	7.82	8.25
	Inside ⅓ of core	7.08	7.08	7.08	7.20
B...	Outside ⅔ of core	7.08	8.92	9.96	10.14
	Inside ⅓ of core	7.08	7.93	8.55	8.48
	2½ hr in Drying Oven				
A...	Outside ⅔ of core	5.32	6.90	7.67	8.55
	Inside ⅓ of core	5.32	5.31	5.31	5.44
B...	Outside ⅔ of core	5.32	13.42	13.03	16.03
	Inside ⅓ of core	5.32	5.66	8.79	12.90
	5 hr in Drying Oven				
A...	Outside ⅔ of core	3.48	7.32	8.40	9.25
	Inside ⅓ of core	3.48	3.48	3.82	4.63
B...	Outside ⅔ of core	3.48	15.43	16.56	17.68
	Inside ⅓ of core	3.48	4.38	5.31	7.48
	10 hr in Drying Oven				
A...	Outside ⅔ of core	1.92	8.15	9.63	11.70
	Inside ⅓ of core	1.92	2.25	2.91	4.26
B...	Outside ⅔ of core	1.92	19.65	19.65	19.95
	Inside ⅓ of core	1.92	2.42	5.34	6.34

Three variables were used: the amount of absolute pressure, the length of time the vacuum was applied to the specimens (pressure time), and the duration of soaking. One item was varied while the other two were kept constant during each series of tests. In the first tests, various pressure times were used at a constant soaking time and a constant amount of absolute pressure, in the second series various soaking times were used at a constant pressure time and absolute pressure, while in the third set of tests various absolute pressures were applied, holding the pressure time and period of soaking constant. Another variable that might have been studied was the time that the vacuum was retained after the specimens were covered with water; how-

TABLE 4.—MOISTURE CONTENT, ONE-HALF IMMERSION METHOD, PER CENT.

Specimen	Part of Specimen Tested	Days of Soaking			
		0	2	4	7
0 hr in Drying Oven					
A...	Top ⅓ of core	7.08	7.08	7.08	7.08
	Middle ⅓ of core	7.08	7.08	7.35	7.35
	Bottom ⅓ of core	7.08	7.82	8.20	8.68
B...	Top ⅓ of core	7.08	8.18	8.45	9.43
	Middle ⅓ of core	7.08	7.96	8.94	9.44
	Bottom ⅓ of core	7.08	9.18	9.68	9.68
2½ hr in Drying Oven					
A...	Top ⅓ of core	5.32	5.32	5.32	5.32
	Middle ⅓ of core	5.32	5.55	5.62	5.67
	Bottom ⅓ of core	5.32	6.97	7.70	8.93
B...	Top ⅓ of core	5.32	10.40	12.78	13.83
	Middle ⅓ of core	5.32	7.41	9.87	11.17
	Bottom ⅓ of core	5.32	16.60	20.03	22.15
5 hr in Drying Oven					
A...	Top ⅓ of core	3.48	3.48	3.48	3.92
	Middle ⅓ of core	3.48	3.48	3.48	4.05
	Bottom ⅓ of core	3.48	6.26	8.90	13.30
B...	Top ⅓ of core	3.48	11.65	14.30	17.35
	Middle ⅓ of core	3.48	9.68	10.65	12.69
	Bottom ⅓ of core	3.48	18.25	22.77	23.38
10 hr in Drying Oven					
A...	Top ⅓ of core	1.92	2.14	2.47	3.24
	Middle ⅓ of core	1.92	1.97	2.47	4.60
	Bottom ⅓ of core	1.92	8.25	12.80	16.10
B...	Top ⅓ of core	1.92	14.10	17.12	20.30
	Middle ⅓ of core	1.92	12.45	15.41	19.10
	Bottom ⅓ of core	1.92	20.00	23.00	25.85

ever, preliminary work showed that this additional time had little influence on the variation in the moisture absorbed.

Immediately after the soaking was completed, specimens were taken out of the jar and their sides blotted with paper towels. Samples for moisture content were removed from the inside and outside of the specimen as in the total immersion method. These moisture samples were then tested to determine the amount of water absorbed.

Discussion of Results

The percentages of water on dry weight basis as was determined for the different methods and variables are shown in Tables 3, 4, and 5.

Total Immersion Method:

Influence of Time of Drying.—The change in the amount of moisture in the outer two thirds of the core (that is, the average of the upper one third and the lower one third) of Shape A specimens is shown in Fig. 2 and the variation of the moisture content in the inner one third of the core of Shape A specimens is shown in Fig. 3. The curves in Fig. 2 (for the outer portion of the cores) indicate definitely that there are greater increases in the amount of water absorbed in the outer portion of the sample as the drying time of the mixture is increased. In other words, specimens dried 10 hr before compaction increased in moisture content in 7 days about 600 per cent, while specimens dried for zero hours increased only about 20 per cent.

The increase in the absorbed water with drying time is not as evident in the inner portion of the specimen. As noted in Fig. 3, specimens formed from mixtures dried for 0 and 2 hr gain almost no water even after 7 days of soaking. After longer periods of drying, the moisture content increased and almost doubled in specimens dried for 10 hr. It might appear from Fig. 3 that specimens dried for 0 hr absorbed more water at 7 days than those made from mixtures dried from 5 to 10 hr. They contained more water, but did not soak in greater amounts of moisture. It must be remembered that this increased water content was present in the mixture when the specimens were molded and thus was not derived from absorption of the water during the soaking procedure.

TABLE 5.—MOISTURE CONTENT, VACUUM-SATURATION METHOD, PER CENT.

Specimen A:							
Pressure time 10 min and absolute pressure 100 mm							
Soaking time, min	0	½	1	2½	5	10	15
Outside ⅔ of core	4.62	9.80	11.45	13.00	13.05	13.55	13.70
Inside ⅓ of core	4.62	8.42	9.17	10.30	11.45	11.95	12.10
Specimen B:							
Soaking time 10 min and absolute pressure 100 mm							
Pressure time, min		0	2	4	8	12	16
Outside ⅔ of core		12.35	13.20	15.75	18.57	19.16	19.76
Inside ⅓ of core		11.70	12.22	14.10	15.46	16.58	16.28
Pressure time 12 min and absolute pressure 100 mm							
Soaking time, min		0	2	4	8	12	16
Outside ⅔ of core		15.50	16.30	16.90	17.42	18.30	18.30
Inside ⅓ of core		11.80	12.20	13.70	14.38	14.64	14.36
Pressure time 12 min and soaking time 10 min							
Absolute pressure, mm				100	200	300	400
Outside ⅔ of core				17.86	17.70	14.94	10.18
Inside ⅓ of core				14.57	9.66	6.64	1.61

Although Figs. 2 and 3 are only shown for Shape A specimens, similar results were obtained for Shape B specimens. Regardless of the shape of the specimens, the amount of water absorbed tends to increase with drying time. In addition, greater increases in moisture develop in the outer portion of the specimens than in the inner part.

Influence of Time of Soaking.—The influence of the time of soaking can also be noticed in Figs. 2 and 3. As the time of soaking is increased, the amount of moisture that will be absorbed in the outer portion of the specimens increases fairly rapidly. However, the influence of the soaking time appears to have little influence upon the amount of moisture gained in the interior of the specimen. Note in Fig. 3 that, except for those specimens which were dried 10 hr before

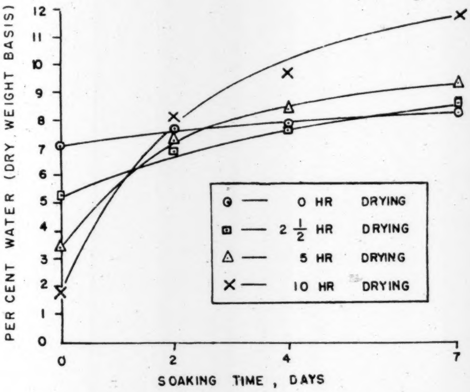

FIG. 2.—Relation of Per Cent Water Content to Soaking Time for Shape A Specimens for Outer Two Thirds of Core; Total Immersion.

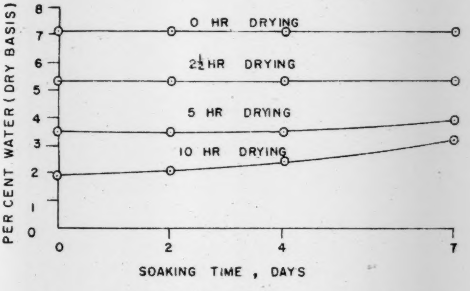

FIG. 3.—Relation of Per Cent Water Content to Soaking Time for Shape A Specimens for Inner One Third of Core; Total Immersion.

compaction, there was little gain in moisture content in the interior of the sample.

There is a tendency for the curves in Fig. 2 to level off as the time of soaking increases. This may be an indication that if specimens are allowed to soak after some optimum time, there may be no further increase in the amount of moisture absorbed by the sample. This point, though, was not definitely established in this study. Seven days was the limitation of the time of soaking and some of the curves had not leveled off by that time.

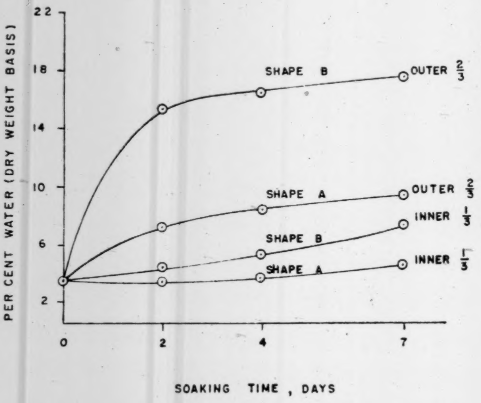

FIG. 4.—Variation in Moisture Content Within Specimen and by Shape of Specimen; Total Immersion, 5-hr Drying.

Variation in Moisture Within the Sample.—Figure 4 shows the variation in the moisture content that took place within specimens dried for 5 hr before compaction as they were allowed to soak for various time periods. Regardless of the type of specimen, the outside portion of the sample increased in moisture content considerably more than the inner part of the specimen. For instance, notice that the outside of Shape B specimens increased in moisture content from 3.48 at the beginning of the test to a moisture content of 17.68 in seven days, an increase of 14.20 per cent moisture content. For the same time of drying, the moisture content of the inner portion of these samples increased from 3.48 to 7.48 per cent, an increase of only 4 per cent. Thus, the moisture content of the outer portion of Shape B specimens increased more than five times their original amount in seven days, while the moisture content on the inside of these same specimens slightly more than doubled in amount. It can also be noticed that for Shape A specimens, the outer portion of the specimens increased in moisture content more than the inside of the specimens, although this increase was not as much as for Shape B specimens. It appears that little or no moisture then reaches the inner portion of the specimens during the first few days of soaking. If the inside portion of the specimen is to increase in moisture content comparable to that of the outer portion, a protracted time of soaking would be necessary.

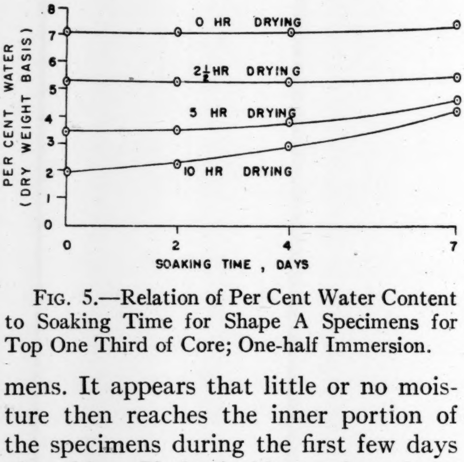

FIG. 5.—Relation of Per Cent Water Content to Soaking Time for Shape A Specimens for Top One Third of Core; One-half Immersion.

In the early stages of their work, Roediger and Klinger (11) obtained results which indicated that "air-blocking" frequently occurred in samples soaked totally immersed. This "air-blocking" occurs when the air is trapped and held in the voids of the specimens by the water pressure. Since the air cannot escape, the water cannot displace it and soaking is retarded. This concept agrees with the results of this study which show a lack of increase in moisture in the inner portions of the sample.

One-half Immersion Method:

In considering the one-half immersion method of soaking, it was believed that the moisture content would vary considerably from the bottom to the top of the specimens. Therefore the moisture determinations were made on the top, middle, and bottom thirds of the core cut from the soaked specimens. Although the major results given in this discussion are for the Shape A specimens, the general trend of results are also applicable to Shape B.

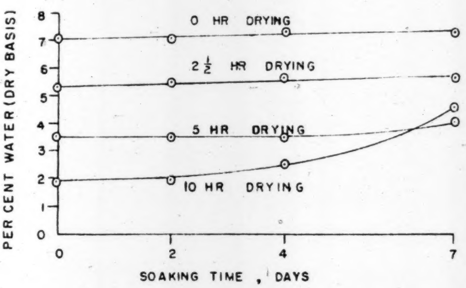

FIG. 6.—Relation of Per Cent Water Content to Soaking Time for Shape A Specimens for Middle One Third of Core; One-half Immersion.

Influence of Time of Drying.—The results of the investigation using the one-half immersed soaking procedure are shown in Figs. 5, 6, and 7. The amount of change in moisture in the upper one third of the core and the middle one third of the core was not influenced appreciably by the time of drying of the mixture before compaction (see Figs. 5 and 6). However, this was not true for the bottom one third of the core which was in the water. In Fig. 7 it can be noted that as the time of drying of the specimens was increased, the amount of water that the bottom portion of the core absorbed tends to increase. In some cases the increase was considerable. For instance, when the specimen was dried for 10 hr before compaction it increased in moisture content in 7 days from 1.92 to 16.10 per cent, an increase of approximately 750 per cent. It is interesting to note, then, that the influence of the time of drying on specimens soaked by the one-half immersion method is similar to that of specimens soaked by the total immersion method.

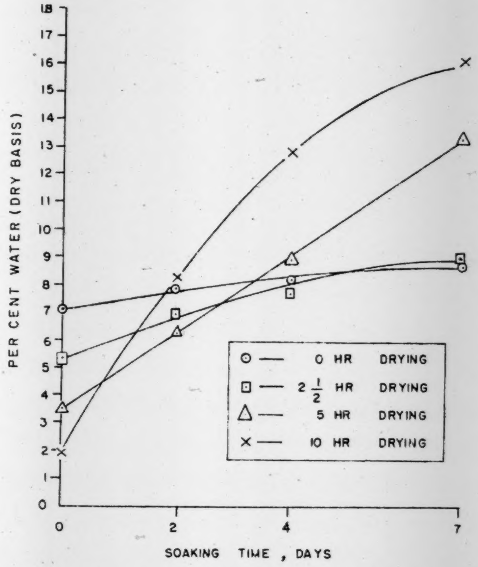

FIG. 7.—Relation of Per Cent Water Content to Soaking Time for Shape A Specimens for Bottom One Third of Core; One-half Immersion.

Influence of Time of Soaking.—Except for those specimens that were dried for long periods of time before compaction, the upper third and the middle third of the specimens increased in moisture content very little, even though the specimens were soaked one-half immersed for 7 days (Figs. 5 and 6). For this method of soaking, however, the bottom portion of the specimens shows a substantial increase in moisture content as the time of soaking was increased. In one case (10 hr drying) there was as much as 14.18 per cent moisture increase. The curves in Fig. 7 also show that this increase tends to level off toward a point of saturation. This phenomenon is not exhibited in the top and the middle portion of the specimen. The increase in water content that does occur in these portions takes place very slowly in comparison with that at the bottom of the specimen.

Variation of Moisture Content in the Specimen.—The variation in moisture content in both Shape A and Shape B specimens after 5 hr of drying with different times of soaking is shown in Fig. 8. Regardless of the type of specimen, there is a considerable difference in the amount of moisture gained in the various portions of the specimen. The largest gain in moisture content is obtained in the bottom one third of all the specimens. In Shape B specimens, the increase in moisture in the top one third is considerably more than in the middle one third. However, little change in moisture was observed in both the top and middle one third of Shape A specimens. The variation in moisture within the specimens can be considerable, as in almost all cases there is at least 100 per cent difference between the bottom and middle portions of the specimen.

At first glance it might seem odd that the upper one third increased in moisture more than the middle one third, since the middle third of the specimen is partially immersed in water while the top one third is completely above the water. Observations made at the time the cores were removed tend to explain this phenomenon. As the water was absorbed into the specimen, it caused the soaked portion of the specimen to turn a darker color than the unsoaked portion. Thus one was able to determine the height to which the water was absorbed and progressed in the soaked specimen during the test. It was noted that the height

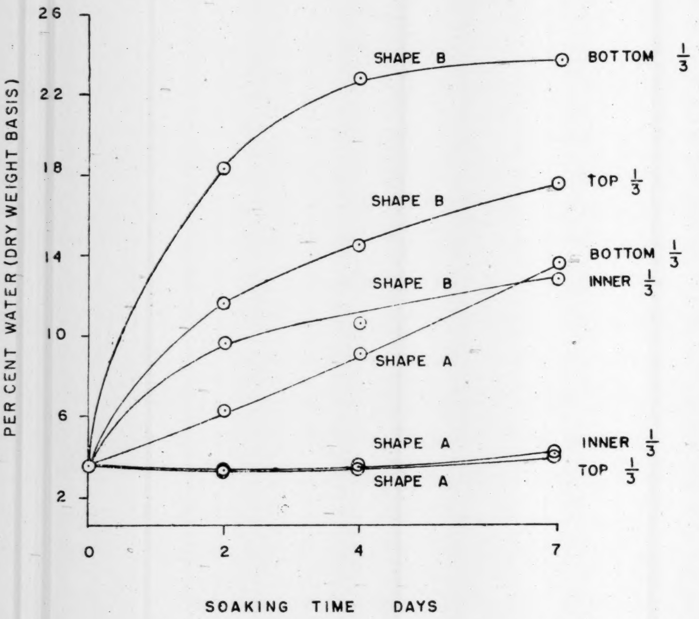

FIG. 8.—Variation in Moisture Content Within Specimen and by Shape of Specimen; One-half Immersion, 5-hr Drying.

of the water soaked was not considerably above the level of the water. It is believed that the major portion of the increased moisture in the top portion of the specimen was derived from the moist atmosphere of the humid room and that little water soaked up from the bottom of the specimen. It is believed that if the moisture in the air was quite low, the amount of increase in moisture in the top one third of the specimen would be considerably lower than that of the middle third of the specimen.

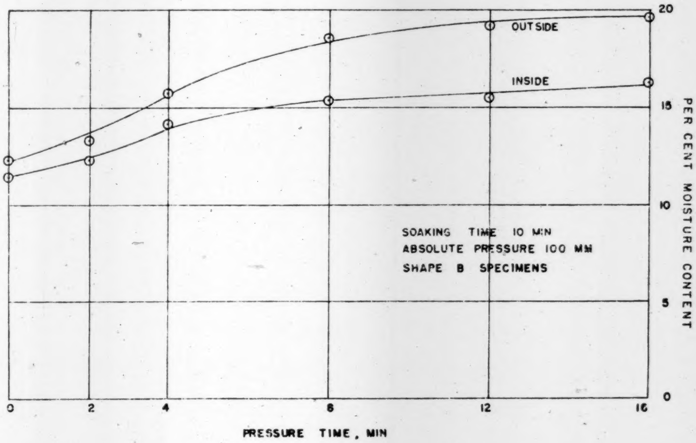

Fig. 9.—Variation in Moisture Content with Pressure Time in Specimens Soaked by the Vacuum-Saturation Method.

Vacuum-Saturation Method:

It was fairly well proved, for the total and one-half immersion methods, that the amount of moisture absorbed by a specimen would increase with the time the mixture was dried in the oven before compaction. Accordingly this variable was not investigated in the study of the vacuum-saturation method. Specimens used in this method were molded only from the mixtures dried in the oven for $2\frac{1}{2}$ hr.

The observation of the "blotter effect" exhibited by the specimen serves to illustrate the mechanism of this vacuum-saturation method of soaking. When a vacuum is applied to a specimen, a portion of the air occupying the void spaces is pulled out and tiny partial vacuums are created. If this vacuum is maintained after the specimens are covered with water, the force pulling the air out of the pores in the specimens hinders the flow of water into the pores produced by capillary action and the small hydrostatic pressure. This retards the amount of soaking that takes place. However, when the specimens are brought back to atmospheric pressure—that is, the top of the water is exposed to atmospheric pressure—the water is forced into the void by the suction of these small vacuums and the pressure of the atmosphere. The samples are saturated within a few minutes. The term "blotter effect" was used to indicate this effect, since it was observed that the surface water was very rapidly absorbed by the specimens upon removal of the specimens from the water after soaking.

As in the total immersion method, it was considered that the top and bottom portions of the specimens would recieve about the same amount of moisture during soaking and that the only variation would occur in the inner portions. Consequently, moisture determinations were made only for the inner and outer sections of the core removed from the soaked specimens.

Influence of Time of Application of Vacuum.—The change in the water content as the vacuum time is varied at a constant vacuum pressure and constant soaking period is presented in Fig. 9. When the vacuum time is increased, the amount of moisture that the specimens absorb tends to increase also. By the time the specimens were completely covered, the moisture content had increased from 5.32 per cent (molding moisture content) to approximately 12 per cent. With an increase in the vacuum time, there is a slight increase in the amount of water absorbed. However, the rate of increase in the moisture becomes less with pressure time and there probably would be little change in water content after about 16 min.

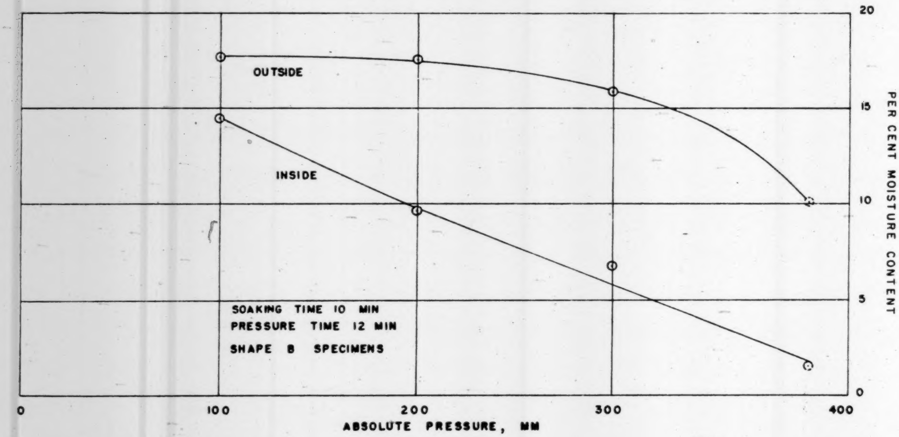

FIG. 10.—Variation in Moisture Content with Absolute Pressure in Specimens Soaked by the Vacuum-Saturation Method.

The amount of moisture in the outer portion of the specimen is greater than that which soaked into the inner portions of the specimens. It is interesting to note that the difference in the amount of water absorbed by the outside and inside was fairly small in the initial stages, up to about 4 min of time of vacuum pressure. But this difference tends to increase with an increase in the vacuum time, although the change was not considerable.

Influence of the Amount of Vacuum Pressure.—The variation in the water content in Shape B specimens as the vacuum is varied, but at constant pressure and soaking time, is shown in Fig. 10. With an increase in absolute pressure—that is, as less vacuum is applied—the amount of moisture that soaks into the specimen tends to decrease. Stated in another way, with greater vacuum, greater amounts of water will soak into the specimen. At the higher absolute pressure (less vacuum) there is considerable difference between the moisture in the outer portions of the specimen and the inner portion. This difference, though, tends to decrease as the absolute pressure becomes less. It is possible that at very high vacuum there will be little difference in the amount of moisture that will soak into the outside and the inner portions of the specimen. It appears, also, that the curves tend to level off at low absolute pressures indicating, possibly, that at pressures less than about 100 mm absolute, there will be little change in the moisture content.

Influence of Time of Soaking.—The results of soaking soil-asphalt specimens at constant pressure time and constant vacuum as the time of soaking is varied is shown in Fig. 11. In the time that it takes for the specimens to be covered by water, the amount of moisture that is soaked into the specimens increases from 5.32 to 11.80 per cent on the inside of the specimen and to 15.50 per cent in the outer portions of the specimen. However, from this initial time on, there is very little increase in the amount of moisture in specimens. Even after 16 min of soaking, the increase in moisture was from 15.50 to 18.3 per cent, an increase of only 2.8 per cent in the outer portions of the specimen. In other words, the specimens will soak up water quite rapidly and then, as the time of soaking is allowed to continue, there will be little increase in moisture in the specimen. Probably after an optimum soaking time there will be no increase in moisture content. Notice that the time of soaking does not appreciably influence the difference in the amount of moisture between the inner or the outer portion of the specimen. The moisture contents do not tend to change and regardless of whether the specimens are soaked for 1 or 16 min the difference in moisture between the outside and the inside of the specimen is approximately the same.

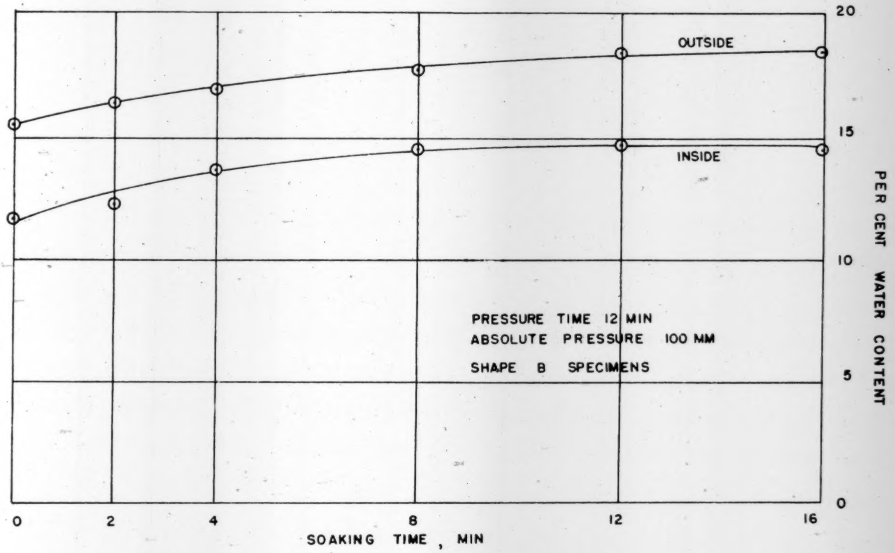

FIG. 11.—Variation in Moisture Content with Soaking Time in Specimens Soaked by the Vacuum-Saturation Method.

Variation of Moisture Content Within the Specimen.—Regardless of the variables studied in connection with the vacuum-saturation method, the amount of moisture in the outer portions of the sample is always slightly greater than the amount of moisture at the inner portions of the specimens. Usually this variation is very small and could be reduced to a minimum by selecting ideal test conditions. For instance, it would appear from the graphs that the minimum difference could be obtained by testing specimens at very low absolute pressure (very high vacuum) and applying this pressure for a relatively short time, say 2 to 4 min, and then covering the specimens and allowing them to soak for only a minute or so. In this case the variation in moisture between the outside and inside would perhaps be almost negligible.

SUMMARY AND CONCLUSIONS

This evaluation of three basic soaking methods has been made in order to obtain a comparison between the moisture-absorbing characteristics of soil-asphalt specimens when soaked by various methods. It was not conducted to determine an appropriate method of test, although the authors feel that the results of this investigation could, after further investigation, lead to the development of a procedure that would be a quick and thorough method of soaking soil-asphalt specimens.

The following conclusions are applicable only to the materials and procedures that were used in these tests:

1. In all specimens there was a difference in moisture content between that of the outer portion of the specimen and the inside of the specimen and, in addition, in the one-half immersion method there was variation within the upper and the lower portions of the specimens. It appears that the greatest variation within the specimen was produced by the one-half immersion method of soaking. Slightly less variation appeared in the specimens that were completely immersed, and possibly very little difference would take place when the specimens were soaked by the vacuum-saturation method under ideal conditions of absolute pressure, time of soaking, and time of application of pressure.

2. Regardless of other conditions, the amount of moisture within a specimen will tend to increase as the time of soaking increases. However, it appears that for most specimens, after a specific length of time of soaking, little increase in moisture within the specimen will take place.

3. Specimens that are dried for long periods of time before compaction will tend to absorb more water than specimens dried for short periods of time. Thus it is desirable to let the material dry for a minimum length of time before compacting in order to control the amount of moisture that the specimens will tend to absorb.

4. Regardless of whether the specimen had a height considerably greater than the diameter of the specimen (Shape B) or whether the height was short as compared to its diameter (Shape A), the variation in moisture content produced within the specimens by the various soaking procedures was similar. Thus, the general results obtained from these various methods of soaking were not influenced appreciably by the shape of the specimen. However, it is interesting to note that the amount of moisture absorbed within the specimens varied with the shape. In most cases the increase in moisture content of Shape A specimens was less than in Shape B specimens. In other words, the triaxial-type specimens will tend to soak up more water and have more variation within them than specimens of the large radius but short height shape (Hveem stabilometer and Marshall specimens).

5. Of the three methods of soaking soil-asphalt specimens, it appears that the vacuum-saturation method could be developed into the most desirable method of soaking soil-asphalt specimens. This method would require considerably shorter periods of time, possibly 15 min as compared with 7 days by

other methods; the soaking would be more thorough and there would be a more equal distribution of the water within the samples.

6. The vacuum-saturation method has considerable advantage over the other methods of soaking in that the amount of moisture that will be absorbed can be controlled. This is due to the number of variables that can be controlled in the vacuum-saturation method—amount of absolute pressure, time of soaking, and time of application of pressure, and so on. If conditions of exceptional amount of moisture are to be studied or if a small amount of moisture is needed, the amount of moisture desired can be obtained by controlling these variables.

7. As noted before, the one-half immersion method and the total immersion method for soaking soil-asphalt specimens produce a wide variability of moisture content within the specimen. These methods of soaking soil-asphalt specimens are not too desirable for producing a sample for standard stability tests. The material would be nonhomogeneous and very erratic results would probably be obtained from specimens soaked under these conditions. It appears, however, that a vacuum-saturation method could be developed that would produce little variation in moisture content within a specimen and, thus, with a homogeneous specimen, the strength evaluations of the soaked material would be quite representative of the material under actual conditions.

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(11) J. C. Roediger and E. W. Klinger, "Soil Stabilization Using Asphalt Cut-Backs as Binders," *Proceedings*, Assn. Asphalt Paving Technologists, Vol. 10, Jan., 1939, p. 1.

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(See Appendix on page 1302.)

APPENDIX

STEPS IN MOISTURE DETERMINATION TEST

1. A 100-g representative sample of the soaked soil-asphalt specimen is placed in a 500-ml round-bottom flask.
2. Approximately 200 ml of purified xylene is added to this 100-g sample and the mixture agitated.
3. The flask and its contents is then clamped to a ring stand. A water trap is inserted in the flask and an Allihn condenser is attached to the top of the water trap. A source of water supply is connected to the condenser at the bottom and an overflow tube attached at the top.
4. A gas burner is placed beneath the flask and the contents allowed to boil at a moderate rate for a period of 5 hr. The condensed liquid collects in the water trap with the water at the bottom and the xylene (specific gravity less than 1.0) on top.
5. The bottom portion of the water trap is graduated in milliliters, and at the end of the boiling period the amount of water present in the original 100-g sample is read directly from the trap.

HEALTH HAZARD FACTORS IN OPERATION OF METAL DEGREASING TANKS*

By E. Elbridge Morrill, Jr.,[1] John M. Blankenhorn,[1] and Paul F. Woolrich[1]

Synopsis

Several factors that must be recognized and fully understood when considering the potential health hazards which may arise from the operation of vapor degreasers employing trichloroethylene or perchloroethylene include the toxicity by inhalation exposures and by skin contact; the permissible standards developed; and the criteria developed for the design, installation, and operation of vapor degreasers to control exposures.

The primary inhalation hazard is from the solvent vapors, but there also may be exposures to gaseous decomposition products of the solvents. There can be exposures to carbon monoxide unless steam or electrically heated coils are used.

There are basic differences in the definitions of threshold limit values (TLV) of American Conference of Governmental Industrial Hygienists and the maximal acceptable concentrations (MAC) established by the American Standards Assn. The TLV is an average weighted exposure for an 8-hr day while the MAC represents a limiting concentration which should at no time be exceeded.

The literature on toxicity of these solvents from both experimental animal studies and human exposures has defined the symptom-concentration relationship. Although acute and chronic toxicities have not been fully resolved, the 1962 TLV of 100 ppm and MAC of 200 ppm give reasonable guide lines to be maintained for the work environment with these degreasers.

The various criteria developed for the design, installation, and operation of the vapor degreasers are outlined, with references to some unfortunate incidents that resulted when the criteria were not met.

The several factors that must be recognized and fully understood when considering the health hazards that may arise from the operation of vapor degreasers using trichloroethylene or perchloroethylene (tetrachloroethylene) include: (1) the toxicity of the solvent, (2) the permissible exposure limits adopted from the available toxicological data, and (3) the pertinent specifications for control measures in the design of equipment, its installation, and its operation.

Health Hazards

The primary health hazard in the operation of a vapor degreaser employing these solvents is exposure to the solvent vapors by inhalation. Under abnormal but not uncommon circumstances workers may also be exposed by inhalation to gaseous decomposition products of the solvents (**1**,**2**).[2] Some of these gases are much more toxic than the solvent vapors themselves. An exposure to carbon monoxide is possible unless steam or electrical heating coils are used in heating the liquid solvents. Prolonged skin contact with these liquid solvents may result in dermatitis, but evidence is lacking that toxic effects occur by way of percutaneous absorption. However, burns of the eyes and skin may occur by exposure to trichloroethylene vapors in high concentrations in a confined space such as inside a degreaser during a cleanout operation (**3**).

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* Presented at the Fourth Pacific Area National Meeting of the Society, Los Angeles, Calif.

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TABLE 1.—CHANGES IN THRESHOLD LIMIT VALUES FROM 1946 TO 1962.

Year	Threshold Limit Values (ACGIH), ppm[a]	
	Trichloroethylene	Perchloroethylene
1946	200	200
1947	150	100
1948–1952	100	100
1953–1960	200	200
1961	100	100
1962	100	100

[a] Parts vapor per million parts of air (by volume).

TLV AND MAC CONCEPTS

When considering these potential health hazards, an understanding of the present-day concept of threshold limit values (TLV) adopted by the American Conference of Governmental Industrial Hygienists (ACGIH) and the maximal acceptable concentrations (MAC) of the American Standards Assn. (ASA) is necessary. There are basic differences in these two standards. Until recently, the definition for these standards were similar, if not identical, but the MAC has now been redefined by the ASA. The MAC represents a limiting concentration or ceiling which should at no time be exceeded. The TLV is an average weighted exposure for an 8-hr day. This definition permits reasonable variations above the value for short durations of exposures provided the time-weighted average exposure does not exceed the TLV for the full 8-hr day (**4**).

There has been considerable fluctuation in the threshold limit values adopted by the ACGIH over the past years. Table 1 points out how the TLV's for trichloroethylene and perchloroethylene have fluctuated since 1946.

In part, this fluctuation stems from the confusion about the definitions of MAC and TLV, particularly in light of the new definition. Some other causes of this fluctuation concern the data on acute and chronic toxicity, particularly as regards the concentration-symptom relationships. Kleinfeld and Tabershaw (**5**) reported that in spite of available experimental and clinical data, there are no clear-cut criteria that would define adequately acute trichloroethylene poisoning, and furthermore, such cases of poisoning have not been satisfactorily resolved. Another probable (possibly more significant) cause of this fluctuation is the uncertainty of potential injury to the liver from exposure to these solvent vapors.

TABLE 2.—SAFE EXPOSURE LEVELS, PPM, REPORTED IN EUROPE.

I.C.I., England	400
USSR	9
Parmegiani (University of Milan)	100
Switzerland	40
Sweden	30

It is of interest to note some of the reported recommended safe exposure levels in Europe to trichloroethylene (Table 2). There is a wide variation in experience with trichloroethylene among **European** workers. The differences in safe limits between European standards and those of the United States have not been satisfactorily resolved at this time.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

Trichloroethylene Toxicity

Shortly after trichloroethylene was first employed as a vapor degreasing agent, reports began to appear in the medical literature (6) concerning its untoward effects. In a study (6) of 284 cases of trichloroethylene exposure including 28 fatalities, the toxic effects reported involved primarily the nervous system. No effects on the liver were described, and their existence was even denied. Subsequent investigators have disagreed with these early reports. They feel trichloroethylene should not be incriminated in all these deaths. Some of the deaths may have resulted from exposure to phosgene or to other agents concommitantly used in the degreasing process. This belief is not without foundation, for there have been instances in the United States of exposures to lethal concentrations of phosgene from vapor degreasing operations (1,2). Adams et al (7), in work with experimental animals, indicate the only toxic effect from single exposures to trichloroethylene vapors is the depressant action on the central nervous system. The ability of even repeated exposure to cause organic injury is of minor importance. Kleinfeld and Tabershaw (5) feel that although most investigators consider the toxic effects of trichloroethylene on the liver to be negligible, there is sufficient reason to be more guarded in their estimation of the potential liver effects.

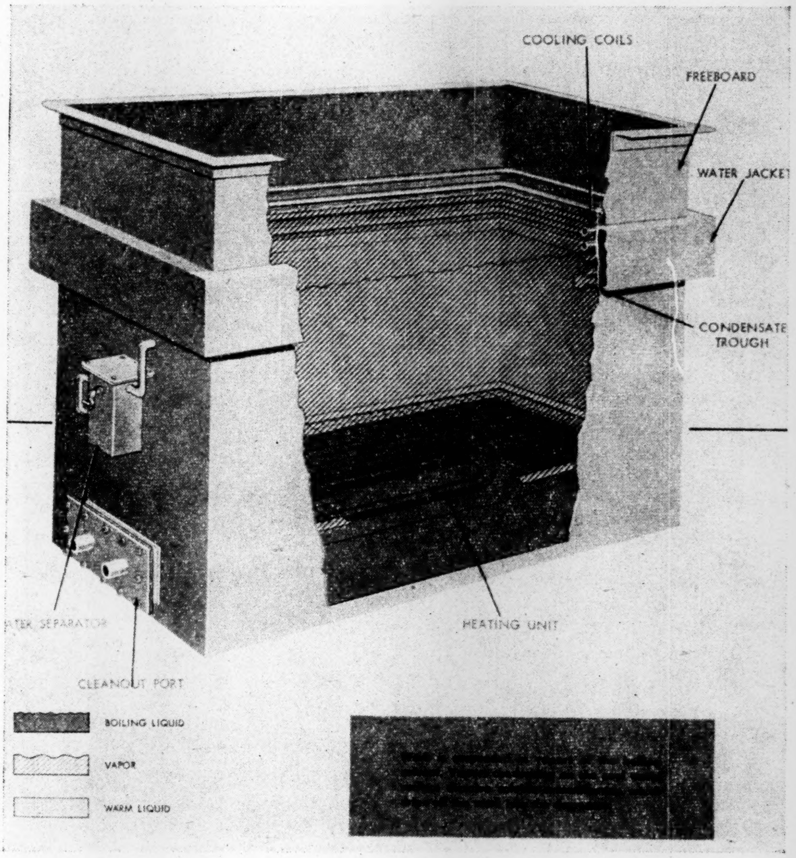
An extensive discussion of the toxic properties of trichloroethylene is given by von Oettingen (8). This solvent is mildly irritating to the skin and somewhat more irritating to the eyes and other mucous surfaces. If excessive concentrations of trichloroethylene are inhaled over extended periods of time, varying degrees of poisoning may result. This solvent is an intoxicant similar to alcohol in the degree and type of inebriation which may result, and it is narcotic; workmen may become addicted to it. There are reports that the use of trichloroethylene produces an intolerance or sensitivity to ethyl alcohol. Overexposure to trichloroethylene vapor is evidenced by a sense of giddiness, possible euphoria, difficulty in walking, and other symptoms of intoxication leading to stupor and unconsciousness. Deaths from acute exposures to trichloroethylene vapors most frequently result from two causes: (1) narcosis to the point of respiratory arrest, and (2) primary cardiac failure (5).

Ordinarily, trichloroethylene (because of its readily noticeable effects of lightheadedness, nausea, and headache) gives ample warning time for an exposed worker to seek fresh air before a serious injury may occur. Furthermore, rapid recovery from acute exposures can be achieved without medical treatment unless exposure has been prolonged and severe. One objective appraisal (9) covering a ten-year period (1948–1957) revealed that 10 deaths and 33 lost-time injuries were reported involving trichloroethylene used in the vapor degreasing process. In addition, there were 58 overexposures requiring medical or first aid treatment with no lost time. Fifteen of these injuries were in 1953. Ten of them occurred during an attempted rescue of another employee trapped in an operating degreaser. Although there were undoubtedly a few injuries that were not reported during this period, the injury frequency rate was of a very low order of magnitude. Entry into a degreaser was the most important single cause of serious injury or death. Furthermore, the injuries from the use of trichloroethylene in the vapor degreasing process resulted from poor safety practices. The above tends to establish that handling trichloroethylene in a vapor degreaser imposes little additional hazard over other industrial processes where no chemical is involved.

PERCHLOROETHYLENE TOXICITY

Perchloroethylene has not been as extensively used as a vapor degreasing agent as trichloroethylene, and consequently has not been studied as thoroughly. However, with the recent advent and widespread use of the coin-operated, dry-cleaning machines on the market today, we find much has been published about its toxicity. Of course, this solvent has been used for many years in dry cleaning establishments, replacing the more toxic carbon tetrachloride. In the earlier reports, before 1940, it was thought that perchloroethylene was relatively harmless, but subsequent work indicates that it should not be so considered. Coler and Rossmiller (10) report a study concerning a small working population in an industry engaged in degreasing operations using perchloroethylene. The investigation was made at this plant because one worker had reported to a local health physician and was having severe gastric hemorrhages. An exploratory operation revealed a definite cirrhosis of the liver and a rupture of an esophageal varix as the cause of the hemorrhage. Two co-workers of this patient had already been seen by the same local physician. These workers were exposed to perchloroethylene vapors twice weekly for a total of 16 hr. Concentrations in the breathing zone for short test runs were measured and found to be in excess of 100 ppm, the MAC level at that time. However, the average readings ranged from 232 to 385 ppm. The workers had complained of headache, nausea, light-headedness, dizziness, tiredness, hangover, and a feeling of intoxication. Some of the workers became unconscious but quickly recovered following removal from the exposure. From this study, the authors concluded that repeated exposure to perchloroethylene in concentrations above the MAC may possibly produce an adverse effect on the liver. It is important to note that the MAC (assumed to mean TLV value) at this time was 100 ppm.

Stewart et al (11) have reported three controlled human exposures to perchloroethylene vapors at levels of about 100 ppm and 200 ppm. There were six subjects in each experiment. Slight irritation and a mild burning sensation of the eye was noted at the level of 75 to 80 ppm after 4 min of exposure. After a few more minutes of exposure, the subjects became unaware of these irritations. In the range of 100 to 120 ppm, a slight irritation and dryness of the palate was noticeable after 4 to 6 min of exposure. At 200 ppm for 6 to 30-min exposures after the start of the experiment, the odor was not unpleasant and Romberg's sign and heel-to-toe tests were normal. Light-headedness occurred 30 min after exposure started at concentrations in the range of 210 to 244 ppm. One interesting observation reported in these experiments was that perchloroethylene had an exceedingly prolonged exponential decay in the postexposure expired air. This decay was characteristic enough to allow a reasonable estimation of the magnitude of exposure. These authors concluded that vapor exposures to perchloroethylene should never exceed 200 ppm because of the rapid onset of light-headedness and therefore the increased risk of accidental injury. Another important point made by the authors is that the perchloroethylene concentration in blood may be used to estimate the magnitude of the vapor exposure only while a subject is being exposed. The rapid drop in the blood concentration of the compound following exposure levels below the sensitivity of the present method of analysis (rapid infrared technique) makes postexposure blood analyses of limited value.

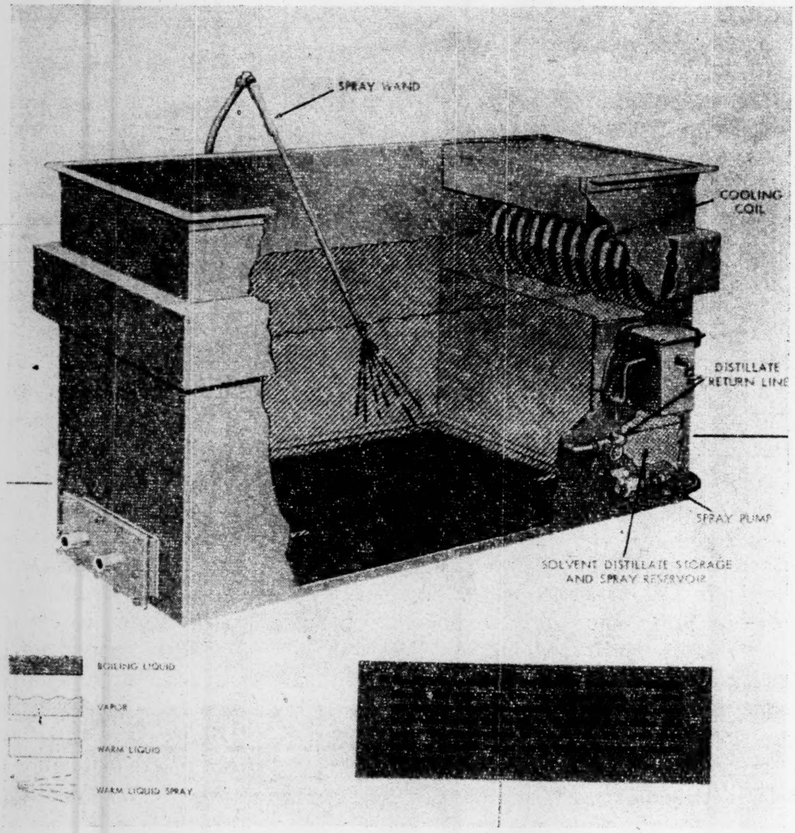


Courtesy of Hooker Chemical Corp.

FIG. 1.—The Hot Vapor Cycle.

Work is suspended in vapors of the boiling solvent. Vapors condensing on the cool metal surface dissolve soluble contamination which drips off the work into the degreaser.

In another article by Stewart et al (12) a case report is presented of an accidental acute exposure to vapors of a solvent mixture containing 50 per cent perchloroethylene and 50 per cent Stoddard solvent. This case did not involve a degreaser, but the worker was cleaning steps with this solvent mixture. One of the authors, wearing an air-supplied respirator, simulated the exposure conditions of the workman and measured the vapor concentrations at 15-min intervals. This study indicated that the worker had experienced an acute exposure to an average concentration of 393 ppm over a $3\frac{1}{2}$-hr period. The concentration of the last half hour was estimated to be 1100 ppm. The authors point out that although a person may have an acute exposure to perchloroethylene vapors there may develop a chronic exposure because of the compound's slow excretion rate. Secondly, liver function may become abnormal during the second and third week after a patient's exposure even though he is completely asymptomatic. The results of their laboratory tests supported their contention that the brief exposure had subjected the liver to a continuous 2½-week insult. The authors conclude that a person receiving an overexposure should not be allowed to experience additional exposures until the expired air concentration is low and all liver function studies are within normal limits.

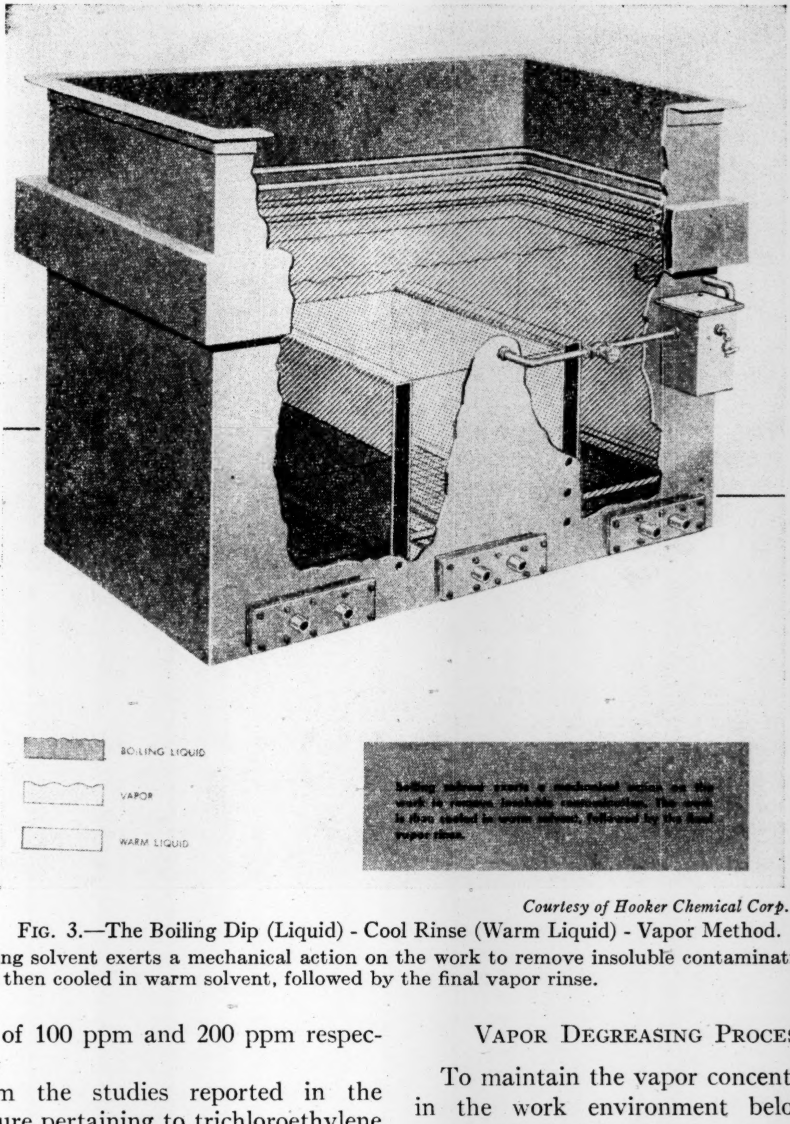

SPRAY WAND
COOLING COIL
DISTILLATE RETURN LINE
SPRAY PUMP
SOLVENT DISTILLATE STORAGE AND SPRAY RESERVOIR
BOILING LIQUID
VAPOR
WARM LIQUID
WARM LIQUID SPRAY

Courtesy of Hooker Chemical Corp.

FIG. 2.—The Warm Flush or Spray-Vapor Method.

Work is suspended in the solvent vapor. Condensation of the vapor on the work stops when the metal reaches the vapor temperature. A warm liquid spray is directed over the work to flush off insolubles and lower the temperature of the metal. The work then receives a final vapor rinse before leaving the degreaser.

In 1952 Rowe et al (13) report from their studies that human subjects experienced central nervous system effects from perchloroethylene vapor concentrations greater than 200 ppm, whereas those exposed to about 100 ppm did not. They likewise recommended a TLV and



Courtesy of Hooker Chemical Corp.

FIG. 3.—The Boiling Dip (Liquid) - Cool Rinse (Warm Liquid) - Vapor Method.

Boiling solvent exerts a mechanical action on the work to remove insoluble contamination. The work is then cooled in warm solvent, followed by the final vapor rinse.

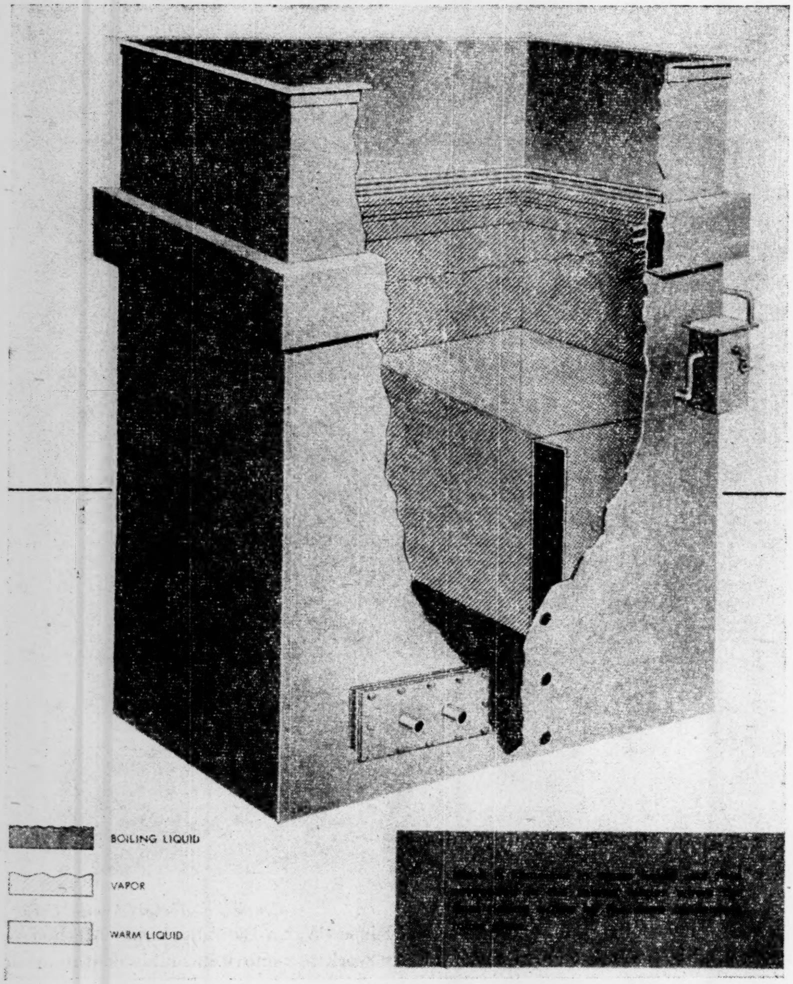
MAC of 100 ppm and 200 ppm respectively.

From the studies reported in the literature pertaining to trichloroethylene and perchloroethylene vapor exposures, much is known about the potential toxicity of the solvents. Although acute and chronic toxicities may not be fully resolved, the present MAC and TL values for these solvents seem to be reasonable guide lines that should be utilized in the design and the operation of vapor degreasing machines.

VAPOR DEGREASING PROCESS

To maintain the vapor concentrations in the work environment below the threshold limit values and maximal acceptable concentrations, the first step in providing control measures is the proper design of the equipment to be used in the vapor degreasing process.

Vapor degreasing is essentially a process of removing oil, grease, wax, and other contaminants from ferrous and nonferrous materials by suspending the



Courtesy of Hooker Chemical Corp.

FIG. 4.—The Cool Dip (Warm Liquid) - Vapor Method.

Work is immersed in warm liquid and then suspended in the solvent vapor where the final rinsing action by the clean condensate takes place.

work into the hot vapor of a solvent, usually trichloroethylene or perchloroethylene. When the metal work at room temperature is introduced or lowered into the vapors inside the degreaser, vapor is immediately condensed on the work. The resulting condensate dissolves the greasy contaminants and drains off the work. Other adhering solids, such as metal cuttings and fines, are also washed off the work by the condensing vapors and are dropped into the degreaser. The action of the solvent continues until the work reaches the temperature of the vapor. At this time condensation ceases and the work is removed cleaned and dry unless there is a carry-out of the liquid solvent entrapped in pocket areas or cavities of the work.

There are four basic cleaning cycles commonly employed in vapor degreasing (14). These cycles are: (1) the hot vapor cycle (Fig. 1), (2) the warm flush or spray-vapor method (Fig. 2), (3) the boiling dip (liquid)-cool rinse (warm liquid)-vapor method (Fig. 3), and (4) the cool dip (warm liquid)-vapor method (Fig. 4). Variations of the basic cycles are often employed for special application. The choice of a particular cycle to be used for any individual installation would depend upon both the nature of the soil (grease, oils, and other contaminants) to be removed and the types of parts to be cleaned.

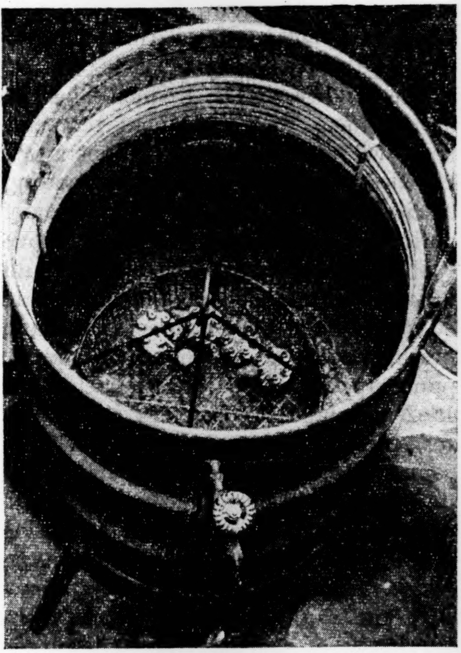


Fig. 5.—Homemade Vapor Degreaser from a 55-gal Drum with Inadequate Free-Board Area Above the Cooling Coils.

Equipment Design Factors

The most essential design factor to prevent escape of the solvent vapors in the work environment is to provide the degreaser with a water-cooled condenser. The condenser consists of a water cooling jacket or a set of pipe coils, or both, extending around the entire inside periphery of the vapor chamber. The condenser or cooling capacity should be sufficient to condense all vapors that are produced which are not otherwise condensed when the cool work is lowered into the degreaser. The condenser should be located at such a depth from the top of the tank as to provide an adequate free-board distance. This is the vertical distance between the lowest point at which vapors can escape from the degreasing machine and the highest normal vapor level. This vertical distance should be at least 15 in. and not less than half the width of the machine. In homemade degreasers which are usually fabricated for special small use, this free-board area is often overlooked (Fig. 5) or cooling coils may not be provided at all. One report comments about an 18-year old worker who was seriously jaundiced from an acute exposure to trichloroethylene vapors from a homemade degreaser which was not provided with cooling coils (15).

Another design factor pertaining to the condenser is that a safety thermostat should be installed above the cooling coils. This safety thermostat should be just above the normal vapor level to shut off the heating elements if the vapor rises above this condensing zone. The thermostat setting should be 180 F for trichloroethylene (boiling point, 189 F) and 240 F for perchloroethylene (boiling point, 249 F) (14). It is important to note that these settings are based upon the boiling point of the solvent; therefore only the solvent for which the tank was designed should be used. Spolyar et al (2) reported an incident in which a worker was exposed to a lethal concentration of phosgene determined to be about 15 ppm when a degreaser originally designed for perchloroethylene was used for trichloroethylene. Although some homemade copper cooling coils had been placed just inside the tank near the top of the degreaser to condense the vapor, they were not in use at the time of the accident. Heating the trichloroethylene above its boiling point, as a result of the thermostat being set for perchloroethylene (higher boiling point), caused the solvent to vaporize rapidly. This rapid vaporization of the solvent lowered the liquid solvent in the degreaser below the level of the electrical heating coils. The authors, in reconstructing the episode, observed that the heating coils became cherry red. Therefore the contact of the high concentration of solvent vapors with the very hot coils resulted in the decomposition of the trichloroethylene vapors to phosgene.

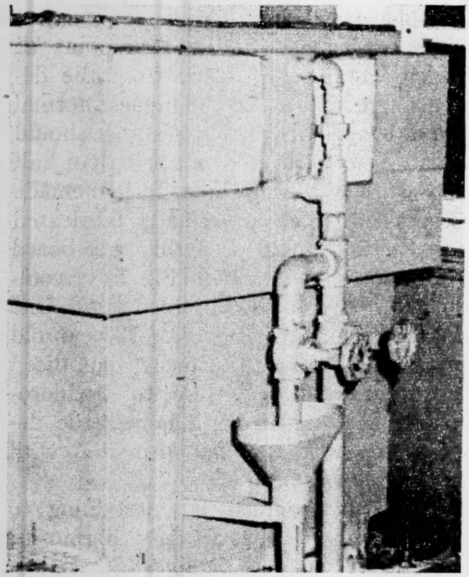


FIG. 6.—Vapor Degreaser Provided with Open "Sight" Funnel to Facilitate Checking Effluent Temperatures.

In addition to the thermostat mentioned above, a thermostat valve (a water temperature control) for the water-cooled condenser should be installed. This valve should maintain the outlet water temperature above room temperature and in the range between 90 and 120 F. The thermometer of this control should be installed between the control valves and the condenser. The spent water should flow through an open "sight" funnel (Fig. 6) before it is discharged into the drain (**14**) to facilitate checking effluent temperatures and to prevent the possibility of back pressure in the condenser. The frequency of the measurements of the effluent temperatures will depend upon the operator's knowledge of the degreaser's operating efficiency and his observations of any noticeable spillage of the vapor above the free-board area. Detection would usually be by odor. The approximate odor thresholds of trichloroethylene and perchloroethylene are 60 ppm and 50 ppm respectively and are well below the 1962 threshold limit values (100 ppm) for these solvents. Absolute reliance cannot always be placed on odor as a means of detection.

It is important that the degreasing machine be of sufficient dimensions in width, length, and depth to accommodate the work to be degreased. Overloading the degreaser may result in unnecessary chronic exposures both in frequency and duration. If the work placed in a large basket which almost fills the cross-sectional area of the degreasing tank is lowered into a tank, there is a piston-like effect. The visible solvent vapors may be noticed escaping. Furthermore, when pieces larger than those for which the degreaser was designed are lowered into the bottom of the tank, employing the boiling dip cool rinse-vapor method, the liquid begins to cool. As the liquid cools, the control valves increase the heat input. Upon removal of the work to the vapor zone area, the high rate of heat input continues for a short time and the vapor is generated rapidly at the time when the heavy hot work is passing through the condensing zone. The degreased work is now practically at the temperature of the solvent vapor and condensation does not take place on the work; therefore some of the vapors can escape from the tank.

Other design factors of the equipment include the provision for a degreaser cover. It may be one that is hinged if there is provision for sufficient head room above the degreaser, or it may be a type that can be rolled over the degreaser. This cover can be used when objects are to remain in the degreaser for long periods of time. The degreaser should always be covered when not in use whether the liquid solvent is at room temperature or at operating temperatures. Covers should be opened and closed slowly to minimize vapor disturbance. Provision for a water separator is desirable since it will lessen the steam distillation of vapors (commonly called ghost vapors) which are more easily dispelled through the atmosphere than pure solvent vapor. Cleanout ports should be provided to facilitate removing the sludge deposits in the tank.

INSTALLATION REQUIREMENTS

Regarding the installation of a vapor degreaser, there are several points to be considered to prevent vapor exposure levels exceeding either the TLV or the MAC standards. The machine should be located in a large, well-ventilated room away from doorways, windows, circulating fans, or other processes that may produce strong drafts. Such drafts, if over 50 ft per min and directed lengthwise of the degreaser tank, may scoop out the vapors, possibly bringing the over-all environment level above the TLV or MAC value. Degreasers should not be located in pits. It is better to provide a platform around the degreaser. The top edge of the degreaser should be high enough above the working level (36 to 40 in.) to eliminate the danger of an operator accidentally falling into the degreaser. If a pit installation is required, an effective exhaust system should be provided for the pit.

Other industrial operations such as welding, electroplating, battery charging, forging, and heat treating or exposures to open flames, hot surfaces or open electric heaters should not be permitted in the immediate area due to the possible formation of phosgene from the decomposition of the solvent vapors. Ferry and Ginther (1) have shown that the decomposition of trichloroethylene vapors into phosgene is a photochemical action caused by ultraviolet radiation from an inert gas welding arc or similar sources of this radiation rather than an actual contact of the solvent vapor with the heat source. They concluded that this fact is of considerable importance in providing suitable control measures. When a source of ultraviolet radiation is near a vapor degreaser, a partial shield should be provided about the arc source to prevent spread of the radiation. Contact of the solvent vapors or liquid with red hot metallic surfaces or open flames may also produce phosgene and other decomposition products.

OPERATION INSTRUCTIONS

Printed instructions should be made available in the immediate vicinity of a vapor degreaser outlining the operational procedures to follow. Preventive maintenance should be performed before starting the degreaser by checking if the solvent level is above the heating elements at a height specified by the manufacturer. The cooling water should be turned on first and the heating elements second, and the cooling water should be kept on as long as the solvent is vaporizing in the degreaser. Other important factors that should be included in these instructions are the proper rate of entry or removal of parts, the necessity of keeping the spray nozzle well below the vapor zone during spraying, and movement of parts from one compartment to another below the vapor level. The operator should frequently check to determine that the various thermostats are operating properly and that the outlet temperature of the condenser water is in the range of 90 to 120 F. Higher temperatures will not give sufficient condensation of the vapors; with lower temperatures there is a tendency to cause excessive condensation of moisture from the atmosphere. An interlocking limiting control mechanism should be installed that would shut off the source of heat when a stoppage of continuous water flow through the condenser occurs.

If a mechanical hoist is used for manually operated degreasers, a rope should not be used as a hoist because of the solvent "drag-out." The work should not be removed from the degreaser until condensation ceases, indicating that the work has reached the temperature of the vapor.

TANK CLEANING

The solvent in a vapor degreaser will become contaminated with oils and metal chips from the work being cleaned. The efficiency of the solvent is nullified when the contamination reaches 30 per cent. Probably the easiest way to determine when this level of contamination has been reached is to observe the boiling temperature of the solvent. If the temperature reaches 195 F for trichloroethylene or 260 F for perchloroethylene, it is time to change the solvent. An alternate is to use the specific-gravity method. The hydrometer reading for this method for trichloroethylene would be 1.17 and for perchloroethylene 1.22. To clean a vapor degreaser of the sludge deposits, it is often necessary that a worker enter the tank, particularly when cleanout ports have not been provided. Before the tank is entered, the liquid solvent should be drained and the tank thoroughly ventilated. A worker entering the degreaser should always wear an air-supplied respirator. (Chemical cartridge respirators are not satisfactory for this work.) An attendant worker should be nearby and be likewise equipped with an air-supplied respirator in the event of an emergency. Even after a degreaser has been cleansed of the sludge deposits, harmful concentrations of the vapor may still exist inside the tank (16).

VENTILATION CRITERIA

A brief comment should be made about mechanical exhaust ventilation requirements for vapor degreasers. To provide a vapor degreaser routinely with ventilation is an expensive practice because of the cost of the ventilation itself, and the resulting high solvent loss. There are instances where ventilation is the only solution to reduce the worker's exposure to the solvent vapors below the permissible level. However, before such ventilation is recommended, a thorough check should be made to determine whether or not all the criteria have been met for the proper design of equipment, its installation and operation. The use of a check list as published by the New York Department of Labor (17) will be very useful in an evaluation for the needs of ventilation. If ventilation is required to control exposures to the solvent vapors, a "slot" ventilation system should be provided. A canopy hood exhaust system should never be utilized. This system actually increases the hazard by drawing the toxic vapor out of the tank and past the operator's breathing zone (16).

SUMMARY

The new demands for cleanliness in aerospace and electronics industries together with the increasing use of the vapors of volatile solvents in new applications of cleaning make necessary a renewed awareness of potential industrial hygiene problems in relation to design criteria and future development of vapor degreasing equipment.

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SOLVENT VAPOR DEGREASING AS APPLIED TO MISSILE CONTAINERS, CONDUITS, TRANSPORTS, AND HARDWARE

By T. J. Kearney[1]

Synopsis

The role of halogenated solvents in the missile industry has become one of prime importance. Cleanliness levels demanded by the aerospace industry, particularly with regard to hydrocarbon contamination can, in most cases, be accomplished by the liquid or the vapor phase, or both, of a volatile solvent such as trichloroethylene, perchloroethylene, or trichlorotrifluoroethane. Such a system has the advantage of ensuring contact with all significant surfaces, introduces no contaminant and, with properly chosen solvent (usually low-residue trichloroethylene) leaves no residue and is easily purged because of its volatility. It inherently provides a method of test for residual contamination.

The foreword of the Aerospace Industries Assn. Handbook on contamination control (1)[2] states the industry's problem most completely with this statement: "The tremendous energies of liquid rocket propellants combined with the complex nature, fine tolerances, and variety of components in liquid rocket propulsion systems make the control of contamination a matter of great importance."

In general, regardless of the propellant system, whether it includes fluorine, hydrogen, oxygen, nitrogen tetroxide, or hydrazine, it is essential that all oils, greases, and lubricants of a hydrocarbon nature be removed from the components and systems. This includes the engine, plumbing and tankage, the ground support and supply system.

Cleaning System for Tanks

The recommended cleaning system for transport tanks, storage tanks, and propellant tanks, when properly applied, will provide a comparatively simple means for removing all soluble organic contamination such as oils, fats, and greases, and much if not all of the particulate contamination. In almost all cases extended vapor generating cycles can be continued or repeated until both hydrocarbon and particulate contamination is reduced to an acceptable minimum. Samples of the solvent distillate off-fall can be checked for both hydrocarbon residues and particulate contamination to determine if further processing is required. As a final rinse, condensate of pure solvent vapors provides a means of rinsing to produce a result meeting both particulate and hydrocarbon limits.

Another advantage is that aluminum or magnesium having a chemically modified sruface, for example an anodized

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[1] Chief Engineer, Detrex Chemical Industries, Inc., Detroit, Mich.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

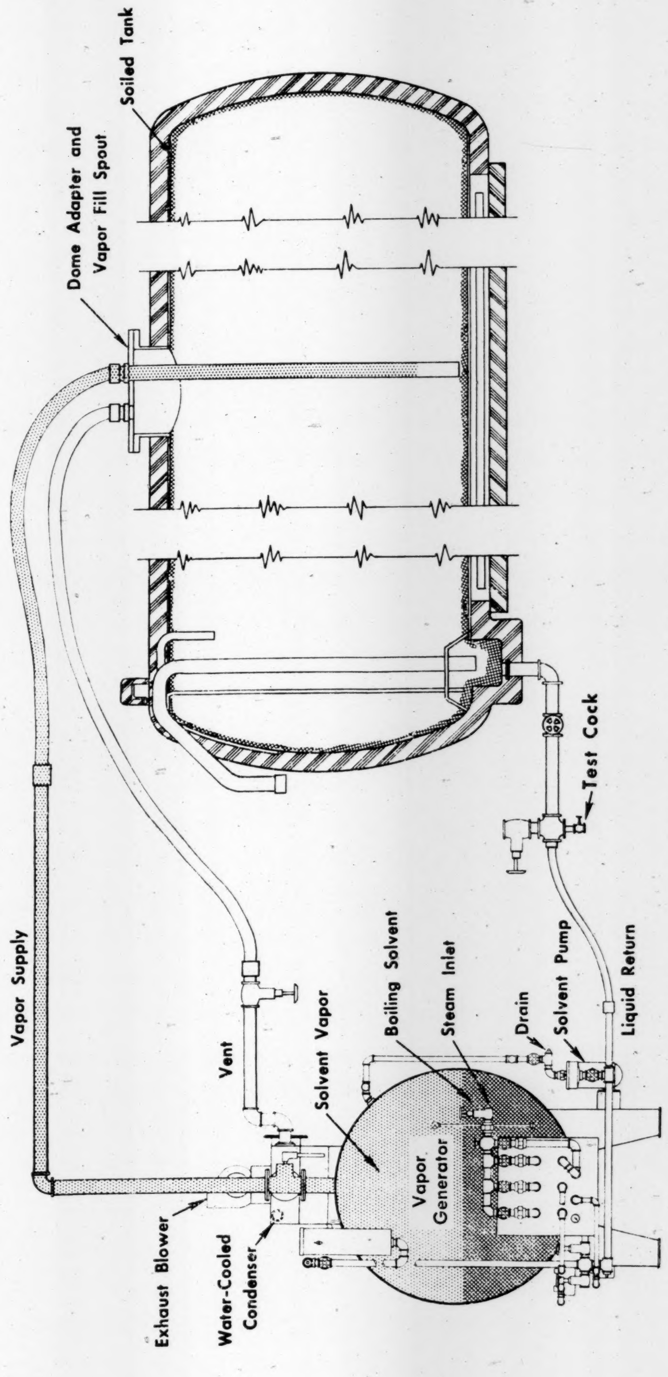
Soiled Tank
Dome Adapter and Vapor Fill Spout
Test Cock
Vapor Supply
Vent
Solvent Vapor
Boiling Solvent
Steam Inlet
Drain
Solvent Pump
Liquid Return
Vapor Generator
Exhaust Blower
Water-Cooled Condenser

FIG. 1.—Schematic Diagram of Vapor Generator Hookup Showing Flow of Vapor from Generator to Tank.

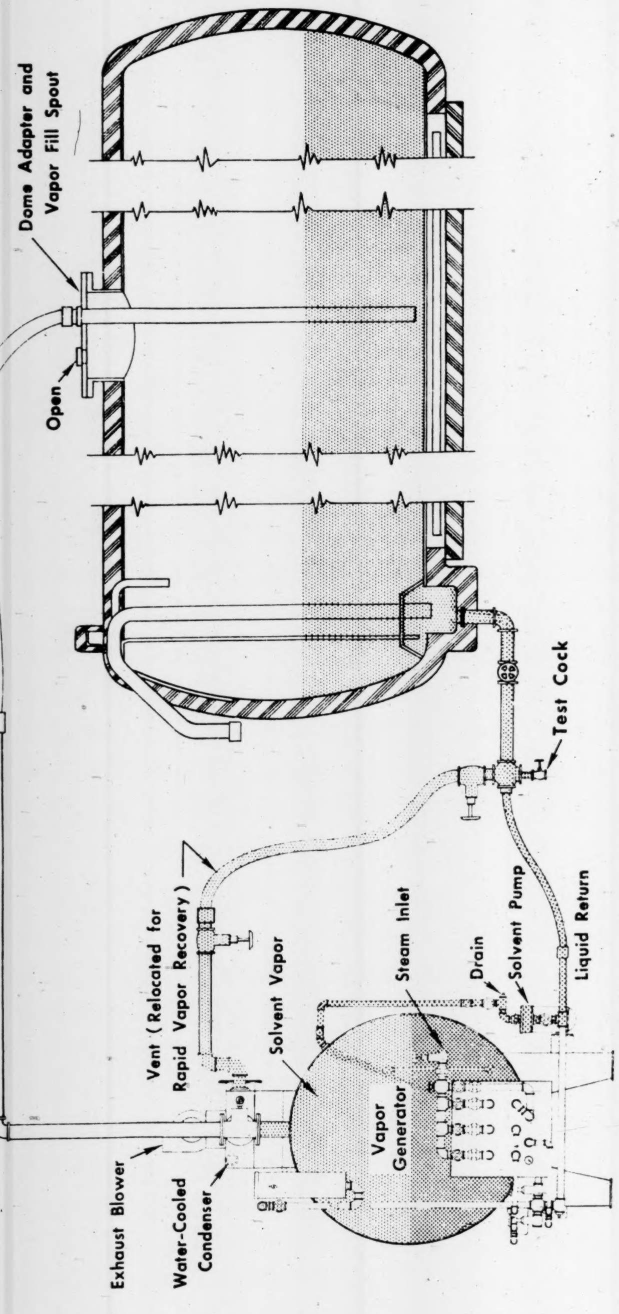
Dome Adapter and Vapor Fill Spout
Open
Test Cock
Vapor Supply
Vent (Relocated for Rapid Vapor Recovery)
Solvent Vapor
Steam Inlet
Drain
Solvent Pump
Liquid Return
Vapor Generator
Exhaust Blower
Water-Cooled Condenser

FIG. 2.—Fast Vapor Removal for Purging Tank After Cleaning.

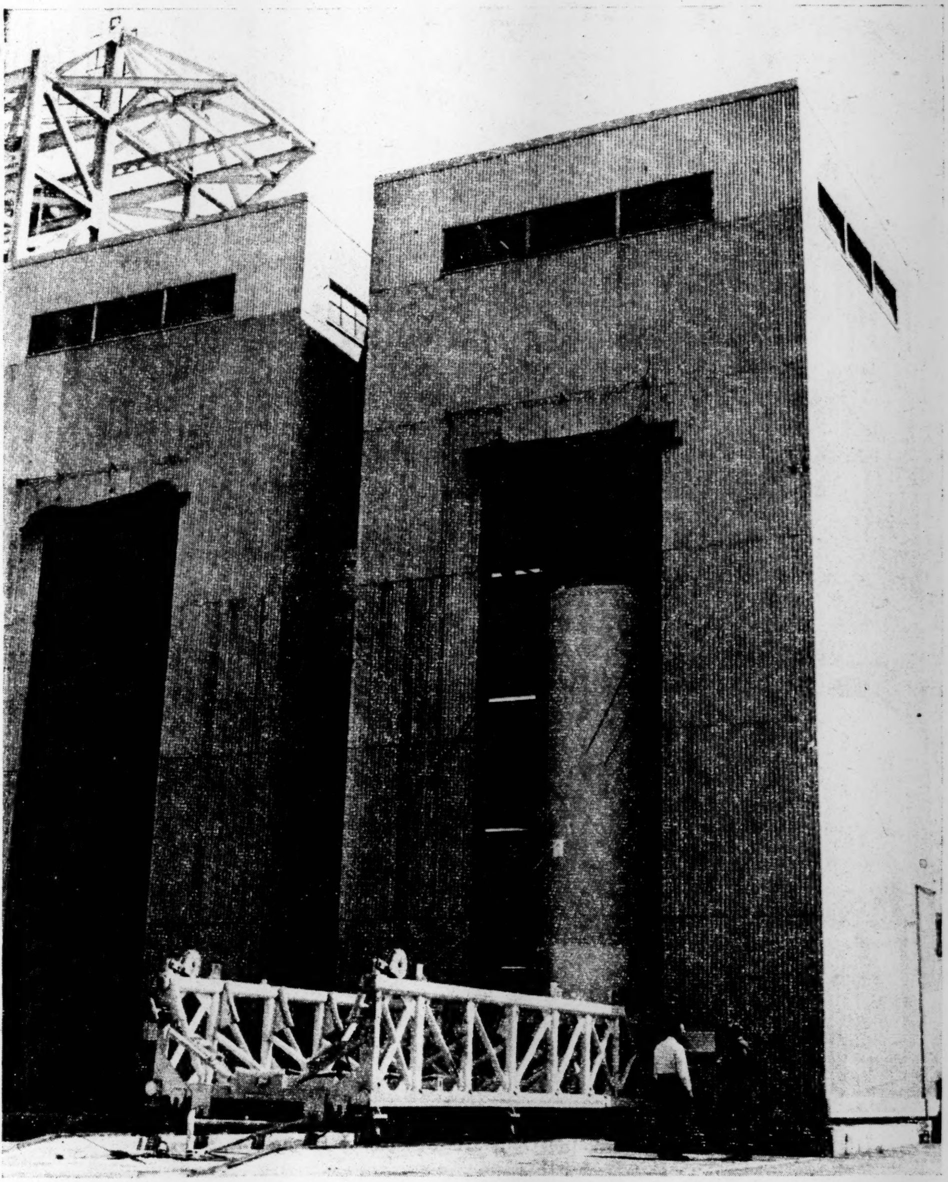

FIG. 3.—Cleaning Installation at a Missile Plant.

coating, can be safely cleaned by this method. The process is not complicated and is shown in the following illustrations.

Figure 1 is a schematic diagram of the vapor generator hookup showing flow of vapors from generator to tank. A dome adapter suited to the construction of the vessel is connected to the vapor supply line, and a flexible line is con-

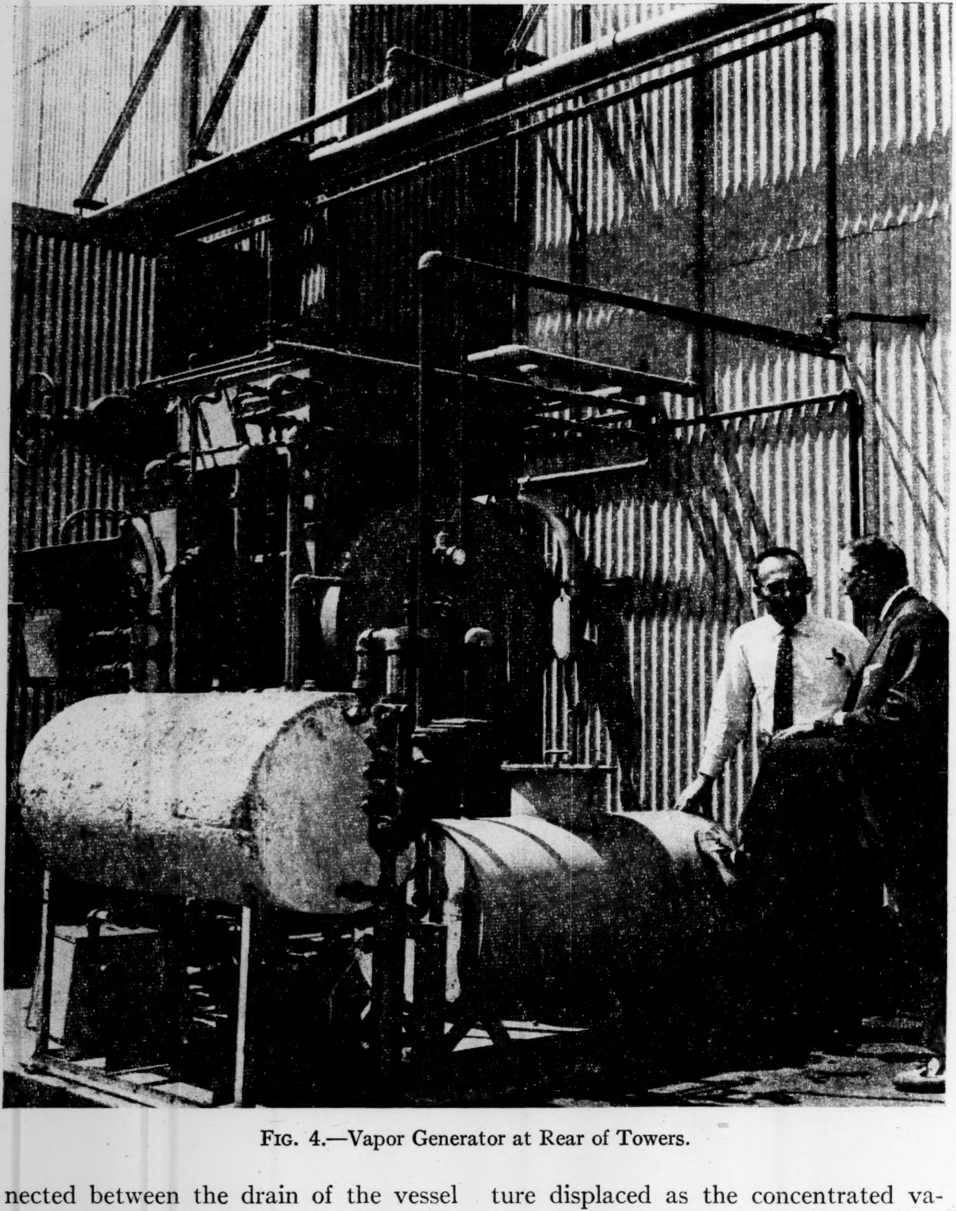

FIG. 4.—Vapor Generator at Rear of Towers.

nected between the drain of the vessel being cleaned and the solvent condensate return pump. A vent is connected between the tank being cleaned and the water-cooled condenser. This condenser removes most of the solvent vapors present in the solvent-air mixture displaced as the concentrated vapors rise in the tank.

The solvent vapors rise in the tank, condensing upon the cooler surfaces, and the condensed solvent and contamination flow down to the drain to the pump suction and are returned to

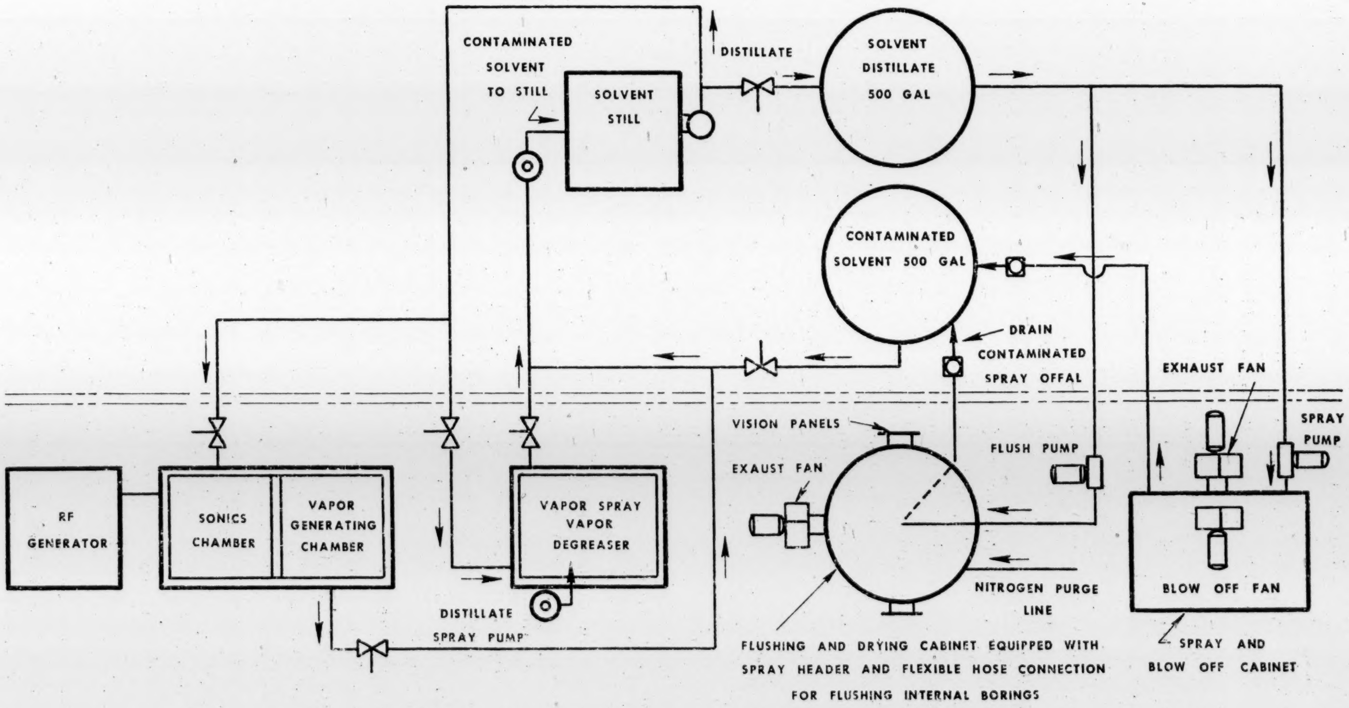
CONTAMINATED
SOLVENT
TO STILL
SOLVENT
STILL
DISTILLATE
SOLVENT
DISTILLATE
500 GAL
CONTAMINATED
SOLVENT 500 GAL
DRAIN
CONTAMINATED
SPRAY OFFAL
EXHAUST FAN
SPRAY
PUMP
VISION PANELS
FLUSH PUMP
EXAUST FAN
RF
GENERATOR
SONICS
CHAMBER
VAPOR
GENERATING
CHAMBER
VAPOR SPRAY
VAPOR
DEGREASER
DISTILLATE
SPRAY PUMP
NITROGEN PURGE
LINE
BLOW OFF FAN
FLUSHING AND DRYING CABINET EQUIPPED WITH
SPRAY HEADER AND FLEXIBLE HOSE CONNECTION
FOR FLUSHING INTERNAL BORINGS
SPRAY AND
BLOW OFF CABINET

FIG. 5.—Cleaning System for Missile Hardware, Especially Hydraulic Components.

All areas and conduit in contact with solvent distillate to be of stainless steel construction. Exhaust fans could discharge to solvent recovery unit.

the vapor generator. The vapors eventually fill the tank and the vessel is completely degreased when saturated vapors flow through the vent line to the condenser.

Samples of solvent condensate off-fall can be taken to check for hydrocarbon and particulate contamination. If the samples do not meet specifications, the unit should be allowed to cool and the cycle repeated. (A normal cycle takes approximately 30 min.)

Figure 2 shows fast vapor removal for purging the tank after cleaning. To remove rapidly the residual solvent vapors (which are approximately $4\frac{1}{2}$ times as heavy as air) and reclaim them, the vent line is removed from the dome adapter and connected to the liquid return line.

Starting the exhaust blower removes the solvent vapors from the vessel and recovers them by passing these vapors through the water-cooled condenser before exhausting them to the atmosphere. Note that all condensed liquid is continuing to flow to the pump and is being returned to the vapor generator. Filtered air is entering the connection at the top of the vessel through the opening previously serving as the vent connection. After satisfactory low limits of contamination are met, the vessel can be purged with nitrogen and sealed.

The entire solvent contents of the vapor generator can be distilled into a storage tank or receiver in 1 to 2 hr, depending upon the size of the unit. The storage tank can also be used as a source of distillate for flushing and rinsing miscellaneous components.

Figure 3 is a cleaning installation at a major missile plant showing the enclosures in which missile fuel tanks are hydraulic tested and vapor degreased.

Figure 4 shows the vapor generator at the rear of the enclosure. The vapor generator system can be applied to the cleaning of: (1) liquid rocket propellant transport tank interiors, (2) liquid rocket propellant ground-based storage tank interiors, (3) liquid rocket airborne propellant tanks, interiors, and (4) insulation annular of cryogenic transport and storage tanks, at the following stages of cleaning: (*a*) before acid treatment for passivating or immunizing, (*b*) after descaling, (*c*) after hydrostatic testing, (*d*) after pickling-neutralizing and rinsing with demineralized water, and (*e*) prior to final nitrogen purging and subsequent sealing.

CLEANING SYSTEM FOR MISSILE COMPONENTS

A comparatively simple and yet extremely flexible cleaning system can be arranged to cope with the cleaning requirements of missile hardware and hydraulic control components. Items that can readily be cleaned in this system include: hydraulic system components, such as pump components, valve and valve components; pneumatic system components; valves and regulators (cryogenic and fuel); gaskets—Teflon, Kel-F, and so on only; and metal expansion joints.

Figure 5 is a schematic arrangement of a hardware cleaning system, installed by a major manufacturer of hydraulic pumps consisting of: (1) a vapor-spray-vapor degreaser, a high-pressure direct connected flushing and purging device to clean interior passages, an ultrasonic degreaser and generator, a spray and blowoff cabinet, and a chlorinated-solvent still. The system also includes solvent distillate and contaminated solvent reservoirs.

It is recommended that all parts be solvent degreased in either the vapor-spray-vapor or the ultrasonic solvent degreaser, depending upon the geometry of the part and the soil to be removed. It is further recommended that all components or parts with internal borings be flushed for 1 min on hydraulic equipment at a flow rate of at least twice the normal flow through the passage being flushed. Following flushing, each passage should be blown dry with gaseous nitrogen. All cleaned components should be completely denuded of solvent and solvent vapors and sealed in polyethylene bags.

No components with elastomeric seals or other materials sensitive to trichloroethylene should be cleaned in this system. Such items could be cleaned in a similar system utilizing trichlorotrifluoroethane or other solvents that do not harm the various elastomers.

SUMMARY

The problem of meeting the various levels of cleanliness in rocket propulsion systems is not simple. The geometry of the part, the type of soil, the materials of construction, and the cleaning fluid must all be considered. The limitations of each cleaning operation must be recognized, and in most cases a specific solution must be used for each commonly recognized contaminant. Experience has shown that new, low-residue or distilled trichloroethylene is an ideal solvent for the removal of soluble organic soils. Trichloroethylene, by dissolving the hydrocarbons, can also remove particulate contamination through entrainment. As a result, it is in most instances the preferred solvent when used in proper equipment for final flushing and rinsing of missile components.

REFERENCES

(1) *Handbook for Contamination Control of Liquid Rocket Propulsion Systems*, Aerospace Industries Assn., March 7, 1960.

(2) R. M. Neary, "Equipment Cleaned for Oxygen Service," International Acetylene Assn., 1959.

(3) "Cleaning Missile Tanks by Vapor Degreasing," *Metal Progress*, Feb., 1960.

(4) "How to Get the Most from Solvent-Vapor Degreasing," *Metal Progress*, Vol. 77, Nos. 4 and 5, April, 1960.

VAPOR DEGREASING WITH TRICHLOROTRIFLUOROETHANE

By R. B. Ramsey, Jr.[1]

Synopsis

The properties of trichlorotrifluoroethane as they pertain to its use in vapor degreasing are discussed and basic considerations for the design, installation, and operation of trichlorotrifluoroethane degreasing equipment employing it are presented. Its performance in vapor degreasing equipment is compared to that of trichloroethylene and perchloroethylene.

Trichlorotrifluoroethane ("Freon" TF solvent[2]) possesses a unique combination of physical and chemical properties which make it an excellent medium for vapor degreasing. Those properties and the basic considerations involved in the design, installation, and operation of vapor degreasing equipment for the utilization of this versatile solvent will be reviewed.

The specifications under which trichlorotrifluoroethane is manufactured and sold by the du Pont Co. are listed in Table 1, together with the tests performed to assure that it meets these specifications. It is the purest, most stable solvent commercially available today for vapor degreasing.

Solvent Properties

Selective Solvency:

Perhaps the most important property of trichlorotrifluoroethane is its selective solvency. It is completely miscible with hydrocarbon oils and most other oils of the silicone or fluorocarbon varieties, and it possesses adequate solubility for the other lubricants and soils generally encountered in vapor degreasing applications. At the same time, it is relatively inert to most plastics and elastomers. Tables 2 and 3 illustrate its superiority over the chlorinated solvents in this respect.

Effect on Metals:

In addition to displaying a general inertness to plastics and elastomers, the solvent is noncorrosive to metals. Articles containing steel, stainless steel, nickel, copper, brass, aluminum, magnesium, beryllium, tin, and zinc are being cleaned safely in this solvent.

A word of caution, however, is in order in regard to the selection of materials of construction for vapor degreasing equipment. Some corrosive attack has been observed on the more reactive metals such as zinc, aluminum, magnesium, or beryllium during prolonged contact with solvent contaminated with free water or certain polar solvents such as ethyl or methyl alcohols. Although a simple test to detect and control such contaminants has been devised (see Appendix I[3]), it is advisable to avoid the use of galvanized

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[2] "Freon" TF solvent is du Pont's registered trademark for trichlorotrifluoroethane.

[3] See p. 1340.

TABLE 1.—SPECIFICATIONS AND QUALITY TESTS FOR "FREON" TF SOLVENT[a]

Specification	Test
1. The product shall be a clear, colorless liquid.	1. Visual inspection of a sample of product.
2. The product shall be passed through a 1-μ filter upon loading into containers.	2. . . . .
3. The product shall be packaged in epoxy-modified, phenolic-lined drums. The drums shall be rinsed with fresh "Freon" TF solvent prior to loading.	3. The quality of the drums is statistically monitored. Some from every shipment are checked. The lining must not craze when dented by a ball peen hammer nor soften when rubbed with cheesecloth soaked in methyl ethyl ketone. Each drum interior inspected visually for flaws prior to loading.
4. The purity of the product shall be 99.8 per cent by weight minimum. (Contains no more than 0.2 per cent of other chlorofluorocarbons.)	4. Gas chromatographic separation with temperature programming through a column composed of 20 per cent by weight of polypropylene glycol on 60 to 80 mesh Chromosorb.
5. The product moisture content shall not exceed 10 ppm by weight.	5. Titration with Karl Fischer reagent using a Beckman Aquameter Model KF-2 automatic titrator.
6. The product residue content (soluble plus insoluble) shall not exceed 2 ppm by weight.	6. Gravimetric measurement of residue from the evaporation of 1000-ml sample.
7. The chloride ion content of the product shall not exceed 0.1 ppm by weight.	7. Titration with mercuric acetate solution using s-diphenyl carbazone as the indicator.
8. The product shall have an acid number of 0.003 mg KOH per g of sample or less.	8. Essentially ASTM Method D 974,[b] modified for use with trichlorotrifluoroethane.
9. The product's boiling point shall be 47.6 C at standard barometric pressure.	9. Gentle refluxing of 250-ml sample in round-bottom flask equipped with thermometer well, electric heating mantle, and water-cooled condenser.

[a] "Freon" TF solvent is du Pont's registered trademark for trichlorotrifluoroethane.

[b] Method of Test for Neutralization Number by Color-Indicator Titration (D 974), 1961 Book of ASTM Standards, Part 7.

TABLE 2.—EFFECT OF VARIOUS SOLVENTS ON ELASTOMERS.

Data derived from tests in which ¼ by 2 in. test strips of various thickness were sealed in glass tubes containing the solvents. Samples were held at temperatures of 75 to 130 F until condition of maximum swell was attained.

Elastomer	Per Cent Maximum Linear Swell in:					
	Trichloro-trifluoro-ethane	Trichloroethylene	Methylene Chloride	Methyl Chloroform	Chloro-form	Carbon Tetrachloride
Thiokol FA[a]	<1 to 1	27 to 48	59 to complete dissolution	11 to 12	91	9 to 14
Buna N	<1 to 1	26 to 36	52 to 65	24 to 33	54	11
Neoprene	1 to 3	43 to 46	37 to 44	35 to 49	43 to 50	35 to 43
Hypalon[b]	1 to 5	36 to 51	39	34 to 46	. . .	33 to 40
Viton A[c]	5 to 6	3 to 4	8 to 10	4 to 9	. . .	1 to 3
Adiprene[d] urethane rubber	7	39	. . .	49	. . .	34
Buna S (GR-S)	9	65 to complete dissolution	26 to complete dissolution	44 to complete dissolution	32	31 to complete dissolution
Natural rubber	17 to 19	41 to 67	34 to 60	58 to 59	45	42 to 44
Butyl (GR-1)	21 to 23	42 to 67	19 to 26	35 to 41	45	50 to 60
Silicone	34 to 36	35 to 49	27 to 31	33 to 37	. . .	35 to 36

[a] Thiokol is a registered trademark of Thiokol Chemical Corp., for its organic polysulfide elastomer.

[b] Hypalon is a registered trademark of E. I. du Pont de Nemours & Co., Inc. for its chlorosulfonated polyethylene elastomer.

[c] Viton is a registered trademark of E. I. du Pont de Nemours & Co., Inc. for its copolymer of hexafluoropropylene and vinylidene fluoride.

[d] Adiprene is a registered trademark of E. I. du Pont de Nemours & Co., Inc., for its polyurethane elastomer.

steel, zinc, and aluminum in the fabrication of degreasers intended for trichlorotrifluoroethane service to avoid the possibility of equipment corrosion due to inadvertent contamination of the solvent during use. In this connection it also should be noted that there is an attendant possibility of work corrosion in such circumstances because of the corrosive nature of the chemical compounds (metal salts) produced by equipment corrosion.

TABLE 3.—EFFECT OF SOLVENTS ON PLASTICS.

Plastic	Solvent											
	Trichlorotrifluoroethane			Perchloroethylene			Trichloroethylene			Methyl Chloroform		
Test Condition[a]	A	B	C	A	B	C	A	B	C	A	B	C
Delrin[b] acetal resin	0[a]	0	0	0	0	0	0	0	0	0	0	0
Zytel-101[b] nylon	0	0	0	0	1	1	0	0	0	0	0	0
Teflon[b] TFE resin	0	0	0	0	1	0	0	1	0	0	0	0
Alathon-7050[b] linear polyethylene	0	0	1	1	6	1	1	1	1	0	2	1
Alathon 9140 or 9141[b]	0	0	2	0	6	2	1	5	2	0	4	2
Poly(vinyl chloride) (unplasticized)	0	0	1	0	4	4	2	4	4	0	4	4
Geon-8700A[c] (high-impact PVC)	...	...	0	...	...	...	...	...	4	...	...	4
Geon-8750[c] (normal impact PVC)	...	...	0	...	...	...	...	...	5	...	...	5
Kralastic[d] ABS polymer	0	0	0	0	4	4	5	6	6	4	5	6
Polystyrene	0	3	6	5	6	6	6	6	6	6	6	6
Lexan[e] polycarbonate resin	0	0	0	2	4	2	4	6	5	5	4	4
Lucite[a] acrylic resin (cast)	0	1	0	1	4	4	5	5	6	1	4	6

[a] Test conditions:
A = 4 hr at 75 F,
B = 4 hr at solvent boiling point, and
C = 100 hr at 130 F.
Effect key:
0. No visible effect,
1. Slightly pliable, no significant swelling,
2. Slightly swollen and pliable or slightly shrunk and softened,
3. Stress cracked and brittle,
4. Swollen, curled and rubbery,
5. Partially dissolved or disintegrated, and
6. Totally dissolved or disintegrated.

[b] Delrin, Zytel, Teflon, Alathon and Lucite are registered trademarks of E. I. du Pont de Nemours & Co., Inc.

[c] Geon is a registered trademark of B. F. Goodrich Chemical Co.

[d] Kralastic is a registered trademark of the U. S. Rubber Co.

[e] Lexan is a registered trademark of the General Electric Co.

Stability:

Trichlorotrifluoroethane is chemically stable. It is unreactive with most chemicals, including nitric acid, acetylene, chlorine, cutting oils, gasoline, and other hydrocarbons. It has been shown to be stable in contact with liquid oxygen when tested for impact sensitivity on an apparatus similar to that described in reference (6).[4]

It is quite stable upon exposure to light and air, and the stabilization with antioxidants and acid acceptors required by most of the chlorinated solvents is unnecessary. As a result there is no loss of inhibitors in the use of trichlorotrifluoroethane or its subsequent recovery from degreaser residues.

Its inherent resistance to hydrolysis, together with its stability in the combined presence of metals and cutting oils, is illustrated by the data shown in Table 4.

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[4] The boldface numbers in parentheses refer to the list of references appended to this paper.

The solvent is quite stable to heat at temperatures far above those likely to be encountered in a well-designed and properly operated vapor degreaser. However, exposure of any halogenated solvent to the high temperatures existing in open flames and exposed electric heating elements may result in its decomposition to toxic and corrosive substances. Care must be taken, therefore, to avoid contact of the solvent and its vapors with open flames and glowing electric heating elements.

TABLE 4.—STABILITY OF VARIOUS SOLVENTS.

STABILITY OF VARIOUS SOLVENTS IN THE PRESENCE OF WATER

Test Conditions.—200 ml of solvent containing 0.1 g of iron powder were refluxed through a water separator (50 ml of water) in a modified Soxhlet extractor for 100 hr. At the end of the test, the increase in acidity in the test system (water and solvent) was determined.

Solvent	Increase in Acidity, HCl equivalent in ppm by weight
Trichlorotrifluoroethane	nil
Inhibited trichloroethylene	9.0
Carbon tetrachloride	15.6
Inhibited methyl chloroform	76.7

STABILITY OF VARIOUS SOLVENTS IN THE COMBINED PRESENCE OF METALS AND CUTTING OIL

Test Conditions.—210 ml of solvent, 45 ml of a chlorinated paraffinic cutting oil, and 45 ml of a sulfurized lard oil were boiled continuously for 24 hr in the presence of 1 g of iron powder and 5 g of aluminum turnings. At the end of the test, the increase in the acidity of the system was determined.

Solvent	Increase in Acidity, HCl equivalent in ppm by weight
Trichlorotrifluoroethane	4.2
Inhibited trichloroethylene	52.7
Inhibited methyl chloroform	965.0
Carbon tetrachloride	1230.0

Flammability:

Trichlorotrifluoroethane has been classed as nonflammable and nonexplosive by the Underwriters' Laboratories (1).

Toxicity:

The solvent is virtually nontoxic. Its life hazard in comparison with various other materials has been studied by the Underwriters' Laboratories with guinea pigs as test animals (1). It has been classified as being much less toxic than methylene chloride, but somewhat more toxic than carbon dioxide. It has been assigned a tentative threshold limit value of 1000 ppm by the American Conference of Governmental Industrial Hygienists (ACGIH), which is the *ceiling value* established by the ACGIH for the atmospheric contamination—with any chemical—to which workers may be exposed day after day without injury to health. It does not necessarily indicate its actual toxicity level (7). The low order of toxicity associated with this solvent is readily apparent, however, when it is recognized that the other principal vapor degreasing solvents have been assigned threshold limit values of the order of only 100 ppm. Its nontoxic nature is probably best attested to by the fact that in all its years of manufacture and use, there is no known instance of toxic effect on personnel handling this solvent.

Although the solvent itself is virtually nontoxic, common sense still has to be exercised in its use. Its vapors, like those of all degreasing solvents, are heavier than air; if they are allowed to accumulate in a confined space, such as a pit, there is a possibility of creating a hazardous condition as a result of air displacement.

Like all degreasing solvents, trichlorotrifluoroethane will extract the natural oils present in human skin, and repeated or prolonged exposure to the solvent and its vapors may produce excessive dryness and subsequent cracking of the skin. Repeated contact with the skin, therefore, should be avoided as much as possible.

TABLE 5.—PHYSICAL PROPERTIES OF VARIOUS DEGREASING SOLVENTS.

	Perchloroethylene	Trichloroethylene	Trichlorotrifluoroethane	Methylene Chloride
Chemical formula	C_2Cl_4	C_2HCl_3	$C_2Cl_3F_3$	CH_2Cl_2
Molecular weight	165.85	131.40	187.39	84.94
Boiling point, deg F	250.2	188.4	117.6	103.6
Vapor density (air = 1)	5.71	4.53	6.47	2.96
Liquid density (at boiling point), lb per gal	12.1	11.35	12.6	10.85
Surface tension at 68 F, dynes per cm	32.3	32.0	19.6	28.2
Latent heat of vaporization, Btu per lb	90	103	63	142
Latent heat of vaporization, Btu per gal	1090	1170	794	1540
Specific heat of liquid, Btu per lb per deg F	0.220	0.233	0.230	0.278
Specific heat of liquid, Btu per gal per deg F (near the boiling point)	2.66	2.64	2.90	3.02
Heat required to bring 1 lb of metal of 0.142 specific heat from 75 F up to vapor temperature, Btu	24.9	16.1	6.05	4.06
Pounds solvent condensed per lb of metal in heating metal of 0.142 specific heat from 75 F up to vapor temperature	0.275	0.156	0.096	0.029
Gallons solvent condensed per lb of metal in heating metal of 0.142 specific heat from 75 F up to vapor temperature	0.023	0.014	0.008	0.003
Radiation and convection losses[a] from degreasers operating at solvent boiling points, Btu per hr sq ft	370	208	63	40
Internal heat losses[b] due to condensation of vapor through contact with liquid surface of wash sump, Btu per hr sq ft	7520	5750	2640	2040
Steam distillation point (at 1 atmos), deg F	190.2	163.8	112.3	100.9
Solvent:water ratio in steam distillation, by weight	5.2:1	13.4:1	101.9:1	66.3:1
Solvent:water ratio in steam distillation, by volume	0.56:1	1.84:1	9.5:1	14.1:1

[a] See Appendix III, p. 1341.

[b] Assumes a heat transfer of 150 Btu per hr sq ft deg F of vapor-liquid temperature difference. Assumed wash sump operating temperatures:
Perchloroethylene, 200 F $\therefore \Delta t = 50.2$ F,
Trichloroethylene, 150 F $\therefore \Delta t = 38.4$ F,
Trichlorotrifluoroethane, 100 F $\therefore \Delta t = 17.6$ F, and
Methylene chloride, 90 F $\therefore \Delta t = 13.6$ F.

Boiling Point:

Because of its low boiling point (117.6 F), vapor degreasing with this solvent is carried out at a much lower temperature than that generally encountered with other degreasing solvents.

Low-temperature vapor degreasing is highly desirable in cleaning many items of electronic hardware since it eliminates the problems that may be encountered with the melting, warping, or thermal softening of polymeric materials or thermal decomposition of the soils being removed.

Low-temperature degreasing is also highly advantageous in those instances where it is desirable to gage the parts or process them further without delay.

When the degreaser is to be located in

an air-conditioned atmosphere, as is so often the case encountered with the cleaning of space-age hardware, low-temperature degreasing is highly desirable to minimize the amount of heat transferred by radiation and convection into the work area. As shown in Table 5, the amount of heat that will be transferred to the surroundings by radiation and convection from a trichlorotrifluoroethane degreaser is only about 30 per cent of that transferred from a trichloroethylene degreaser or only 17 per cent of that emitted from a perchloroethylene degreaser.

The brief time intervals required for degreaser start-up and for bringing the work up to vapor temperature, are, of course, attractive operating features of low-temperature degreasing which permit maximum use to be made of the degreasing equipment. The short time required for bringing the work up to operating temperature also assists in minimizing and eliminating the drag-out losses that occur if temperature equilibration is not attained before the work is removed from the degreaser.

The low boiling point of this solvent also minimizes the possibility that the contaminants will be co-distilled in the degreaser, thereby assuring that the vapor rinse will be effected with solvent possessing a high purity. Because of the same consideration, solvent of high purity can be recovered readily from degreaser residues by distillation in stills of the most elementary design.

Vapor Density:

Of all the solvents employed in vapor degreasing work, trichlorotrifluoroethane possesses the greatest vapor density, having a vapor density roughly 6.5 times that of air at standard gas conditions and a vapor density 1.7 times that of trichloroethylene and 1.4 times that of perchloroethylene at atmospheric boiling conditions (Table 5). This property makes it possible to maintain a highly stable, definite vapor line in the machine, which is less subject to upset from drafts and operating disturbances.

Surface Tension:

The solvent possesses the lowest surface tension of all of the commonly used vapor degreasing fluids. As shown in Table 5, its surface tension of 19.6 dynes per cm is only 60 to 70 per cent of that of the chlorinated solvents. This low surface tension enhances solvent penetration into blind holes and recesses in the work being cleaned.

A low surface tension also is essential in the degreasing of plastic materials which themselves possess low critical surface tensions. The degreasing fluid will not wet the surface of such materials unless its surface tension is less than that of the critical surface tension of the plastic. Of the commonly used vapor degreasing fluids, trichlorotrifluoroethane is the only one that possesses a surface tension low enough to wet the surface of tetrafluoroethylene resin (Teflon[5]).

EQUIPMENT DESIGN

The selection and design of a vapor degreaser for use with this solvent are best accomplished by consultation with reputable manufacturers of such equipment. The size and type of machine selected will depend upon the size and shape of the work, the methods of work racking and handling, the expected hourly production rate, the amount and type of soil normally present, the degree of cleanliness required, and the type of utility services available. The various basic types of vapor degreasing equipment are well described in the literature (**2,3,8**), and will not be discussed further in this paper.

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[5] Teflon is a registered trademark of E. I. du Pont de Nemours and Co.

The equipment employed with trichlorotrifluoroethane generally is somewhat similar in appearance to that used with the chlorinated solvents. There are, however, certain basic differences that should be considered.

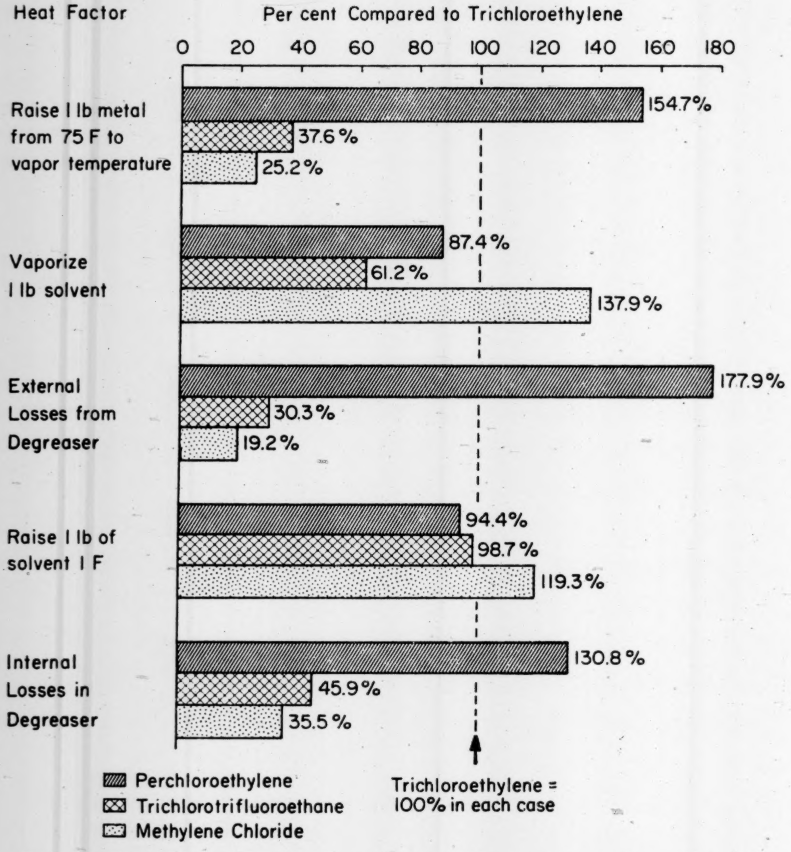


FIG. 1.—Comparison of Heat Considerations of Various Degreasing Solvents.

Heat Requirements:

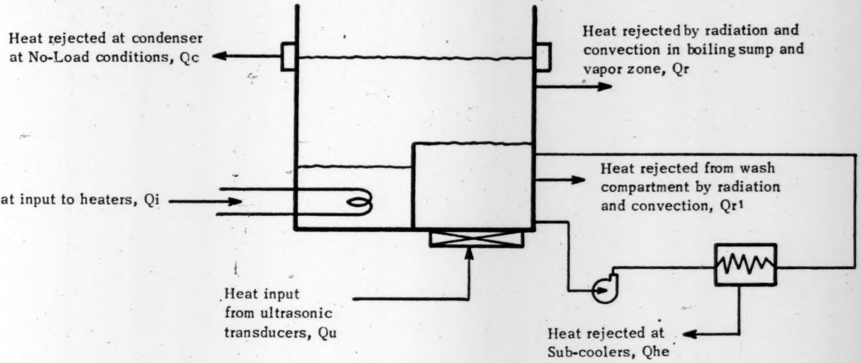
A major point to consider in the design of any degreaser is the heat requirement. Following the selection of the type and tentative size of the proposed unit, the designer's first task will be to develop a heat balance which will take into account the heat required to bring the work load up to degreasing temperature, the heat required for internal refluxing of solvent, the heat losses by radiation and convection, and the sensible heat removal required for control of wash sump temperatures, plus any heat considerations introduced by the inclusion of ultrasonic equipment in the degreaser wash sump.

The magnitude of the various heat considerations will vary quite markedly for the same work load, depending upon which solvent is selected for the degreasing medium. The thermal properties of the common degreasing solvents are listed in Table 5 and graphically compared in Fig. 1.

The full effect of these differences in thermal properties is illustrated by the

TABLE 6.—COMPARISON OF THE PERFORMANCE OF A TWO-DIP VAPOR DEGREASER WHEN OPERATING WITH VARIOUS SOLVENT MEDIA.

BASIS.—Machine contains two compartments each approximately 20 by 20 by 15 in. deep. Wash compartment contains 26 gal of solvent. Vapor zone above wash compartment is 15 in. deep. Boiling sump contains electrical heaters of 12-kw capacity. Wash compartment equipped with 500-w ultrasonic transducer.


Heat rejected at condenser at No-Load conditions, Qc
Heat rejected by radiation and convection in boiling sump and vapor zone, Qr
Heat rejected from wash compartment by radiation and convection, Qr¹
Heat input to heaters, Qi
Heat input from ultrasonic transducers, Qu
Heat rejected at Sub-coolers, Qhe

	Solvent					
	Trichloroethylene		Perchloroethylene		Trichlorotrifluoroethane	
Vapor temperature, deg F	188.4		250.2		117.6	
Wash sump temperature, deg F	150		200		100	
	Btu per hr	per cent of total	Btu per hr	per cent of total	Btu per hr	per cent of total
Heat balance:						
Input:						
Qi	40 944	96	40 944	96	40 944	96
Qu	1 706	4	1 706	4	1 706	4
Total	42 650	100	42 650	100	42 650	100
Output:						
Qc	17 540	41	8 220	19	29 831	70
Qr	4 380	10	7 960	19	1 357	3
Qr'	1 130	3	2 170	5	568	1
Qhe	19 600	46	24 300	57	10 894	26
Total	42 650	100	42 650	100	42 650	100
Solvent circulation rate:						
To wash compartment, lb per hr	338		349		597	
To wash compartment, gal per hr	30		29		47	
Wash sump turnover, times per hr	1.15		1.11		1.82	
Work load capacity,[a] lb per hr	545		165		2 470	
Miscellaneous heat loads:						
Internal loss due to condensation at liquid surface in wash sump, Btu per hr	16 000		20 900		7 340	
Sensible heat removal from condensates entering wash sump, Btu per hr	3 024		3 864		2 416	

[a] Assumes 50 per cent of *Qc* available to heat metal of 0.142 specific heat from 75 F to vapor temperature.

data of Table 6. Here a theoretical comparison is made of the performance of the same degreaser when operating with each of the three most widely used degreasing solvents.

Particularly noteworthy is the much higher work load capacity of the machine when employing trichlorotrifluoroethane, indicating the greater availability of useful heat associated with the employment of this solvent. It should be noted that a high heat availability at the condenser is a very desirable condition in a vapor degreaser since it minimizes the possibility of vapor zone collapse during the work introduction period. Should vapor zone collapse occur in a vapor degreaser, air naturally will rush into the machine to fill the void so created. The subsequent expulsion of this air during the reestablishment of the vapor zone occasions, siolvent losses of substantial proportions and for this reason vapor zone collapse represents a highly undesirable situation which should be avoided.

In connection with the condenser heat load, it should be remembered that the heat transfer area required at the condenser is proportional to the heat load and inversely proportional to the mean temperature difference between the solvent vapors and the cooling medium.

In comparison with trichloroethylene and perchloroethylene machines of equivalent heat input capacity, it is obvious that trichlorotrifluoroethane degreasers will require somewhat larger condensers since they operate at a smaller condenser temperature differential with much larger condenser heat loads.

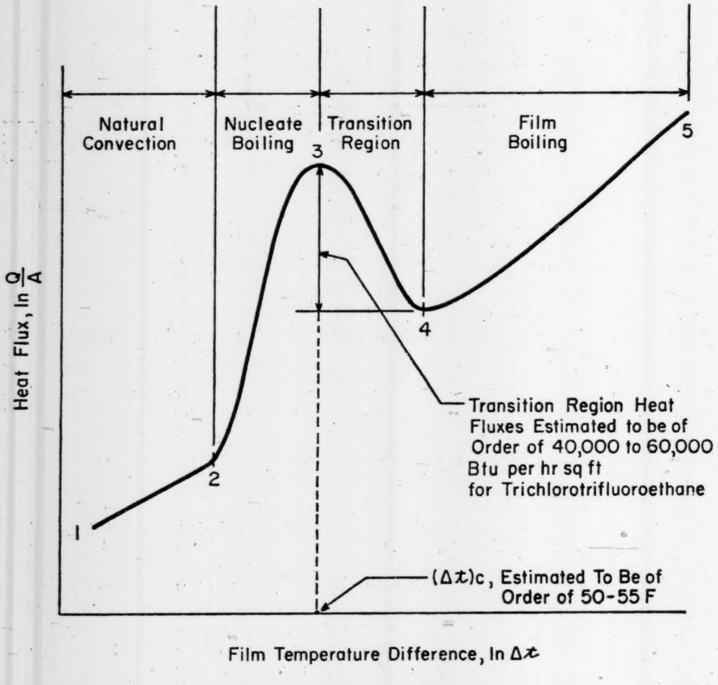


FIG. 2.—Regions of Boiling Heat Transfer.

Heating Facilities:

Trichlorotrifluoroethane degreasers can be heated by any of the conventional methods employed for trichloroethylene or perchloroethylene degreasers. However, the low operating temperature of a trichlorotrifluoroethane degreaser permits consideration of another method of heating employing hot water at 160 to 180 F as the heat source. It can thus be used in those plants where building heating is done with hot water systems or where waste heat in the form of condensates from other operations is available.

Where steam heating is employed, steam at pressures of 0 to 10 psig is adequate for low-temperature vapor degreasing whereas trichloroethylene machines require 10 to 15 psig steam, and perchloroethylene units require 50 to 60 psig steam. Utilization of low-pressure waste steam is therefore also a definite possibility with trichlorotrifluoroethane.

To avoid excessive electric heater skin temperatures which may cause decomposition of either the solvent or the contaminants present in the boiling sump, one should take care to select heaters having a heat flux that will ensure that the heat transfer takes place in the nucleate region of boiling (see Fig. 2).

The Kutateladse correlation (4) (see Appendix II[6]) predicts a peak heat flux for the nucleate boiling of trichlorotrifluoroethane solvent of 65,300 Btu per hr sq ft (133 w per sq in.), which appears to be confirmed by the work of Mesler and Banchero (5) in which they encountered film boiling at heat fluxes of 42,000 to 60,000 Btu per hr sq ft (85.5 to 122 w per sq in.). However, in order to guarantee that the electric heaters will be operating in the nucleate region with this solvent, the heat flux of such equipment is generally limited to maximum values of 25 to 30 w per sq in.

Moisture Control:

Aside from possible adverse effects on the cleaning process or on the articles being cleaned, water in any degreaser is undesirable because of the solvent losses that occur as a result of its presence.

For all practical purposes, water is insoluble in all of the commonly used degreasing solvents. Therefore its accumulation in a degreaser results in a two-phase liquid mixture, with water constituting the upper phase because of its lower density. In the boiling sump of the degreaser such mixtures co-distill, and the amount of each constituent (solvent and water) in the resulting vapors is proportional to the vapor pressures of the pure components at the distilling temperature. Because of their lower vapor density, such mixtures diffuse out of the machine into the surrounding atmosphere much more rapidly than do the heavy, pure solvent vapors.

Because of the lower operating temperature, the control of moisture in trichlorotrifluoroethane machines is not as critical as it is in trichloroethylene or perchloroethylene machines. The solvent - water vapor mixture generated by the co-distillation of the two-phase system in the boiling sump contains only 10 volume per cent of water in a trichlorotrifluoroethane machine, while such mixtures in trichloroethylene and perchloroethylene machines contain, respectively, 35 and 64 volume per cent of water. In the case of trichloroethylene, the reduction in vapor density occasioned by the presence of free water in the degreaser amounts to about 30 per cent, while with perchloroethylene, it amounts to about 57 per cent. With trichlorotrifluoroethane the reduction is only 9 per cent, and the water-saturated vapors still possess a density approximately 5.9 times that of air, which is, of course, a greater density than that possessed by the undiluted vapors of pure perchloroethylene.

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[6] See p. 1340.

Other than being carried into the degreaser on the objects being cleaned, the principal source of moisture introduction is the condensation of atmospheric moisture from the air in contact with the condenser. For minimum atmospheric condensation, the condenser should be operated at a temperature above the dew point of the room in which the degreaser is located. Generally this is accomplished by throttling the condenser water flow so as to yield an exit water temperature from the condenser of about 90 F. On many machines, the throttling is effected by placing a thermal bulb in the exit water line to sense the exit water temperature and actuate a control valve in the condenser feedwater line.

TABLE 7.—SOLUBILITY OF WATER IN TRICHLOROTRIFLUOROETHANE.

Temperature, deg F	Solubility, ppm by weight
−30	6
−20	8
−10	11
0	15
10	20
20	26
30	34
40	44
50	55
60	70
70	90
80	113
90	140
100	168
110	210

As in the case with trichloroethylene and perchloroethylene machines, a condensate subcooler and water separator in the condensate return system are often very desirable auxiliaries, particularly on the larger units.

If an appreciable accumulation of moisture is expected as a result of carry-in on the objects being cleaned, a trough to collect the overflow from the wash sump and divert it through a water separator on its way to the boiling sump may be desirable. In some moisture-sensitive cleaning operations where it is necessary to keep the moisture content of the solvent below saturation limits (Table 7), an adsorptive dryer filled with silica or alumina gel has been employed in a recirculation system connected to the wash sump to dispose of the moisture introduced to the degreaser.

Condensing Facilities:

The design of condensing facilities for use with trichlorotrifluoroethane machines will generally be similar to those employed with the chlorinated solvents, but for the reasons previously discussed, will be somewhat larger in size.

As is the case with all degreasers, a portion of the condensing surface should be an integral part of the four outer walls in order to minimize convective currents in the machine.

It also is good practice not to connect the condenser water outlets directly to sewer lines but instead to drain them into an open sight device, such as a funnel. This prevents the possibility of back pressure causing subsequent condenser failures. A funnel connection also provides a convenient means of checking water flow and water outlet temperature.

Solvent Loss Control:

The principal sources of solvent loss from a degreaser are the following: (1) diffusion due to mixing of solvent vapor with air, (2) diffusion due to mixing of solvent vapor with water vapor, (3) drag-out of liquid solvent from the degreaser, (4) losses due to leaks and spills, (5) loss of solvent in discarded degreaser sludges, and (6) losses due to improper use.

In a perfectly stagnant atmosphere, the theoretical loss due to diffusion of trichlorotrifluoroethane vapors through a 16¼ in. layer of air is approximately 0.126 lb per hr per sq ft of vapor-air interface, as compared to losses of 0.075 and 0.084 lb per hr per sq ft for trichloroethylene and perchloroethylene respectively. The actual losses under "idling" conditions, however, for operation with trichlorotrifluoroethane may be no greater, and could conceivably be even less, than those encountered with trichloroethylene and perchloroethylene because (1) the greater vapor density of trichlorotrifluoroethane (1.4 to 1.7 times that of perchloroethylene and trichloroethylene respectively) makes it easier to approach more nearly ideal stagnant conditions at the vapor-air interface, and (2) because of the lower operating temperature of a trichlorotrifluoroethane machine, thermal updrafts at the exterior walls are not as severe as they are with trichloroethylene or perchloroethylene.

However, it should be recognized that the theoretical diffusion losses may be multiplied several times by disturbances at the vapor-air interface caused by drafts from windows, doors, unit heaters, or exhaust fans adjacent to the degreaser, by the improper operation of spray lances at or above the vapor-air interface, by rapid work introduction or withdrawal, by improper work handling, or by the introduction of excessive work loads.

It is evident from the foregoing considerations that the biggest factors in minimizing vapor-air diffusional losses probably are proper sizing of the degreaser initially, proper installation subsequently, and finally, rigorous adherence to good operating practices. For optimum performance:

1. The degreaser should be selected to have an adequate heat input to handle the normal work load.
2. The location of a degreaser should be carefully selected to place it in as draft-free an area as possible.
3. To avoid a "piston" action upon the vapor zone during work insertion or withdrawal, the bottom area (length times width) of the basket or work load should not exceed one half to two-thirds of the work area.
4. Work loads should be handled in and out of the degreaser at slow, uniform speeds, preferably on the order of 5 to 10 ft per min.
5. All spraying operations should be conducted as far below the vapor level as possible, and care should be taken to avoid ricocheting of sprayed solvent above the vapor level.
6. All transfers of work from one compartment to another in a multicompartment machine should be made with the work below the vapor line to minimize vapor-air interface disturbances. (Multicompartment machines, therefore, must contain a vapor zone deep enough to make such transfers possible.)
7. Covers should be closed when a degreaser is not in actual use.
8. Covers should be opened and closed slowly to avoid the creation of atmospheric disturbances within the machine.

In connection with items 7 and 8, it generally is inadvisable to shut a degreaser down between intermittent use periods, since the vapor losses encountered in expelling air from the machine to reestablish the vapor zone may be considerably greater than those incurred by allowing the machine to "idle" with the covers on.

There are several features that can be designed into a degreaser installation which also will help to minimize vapor-

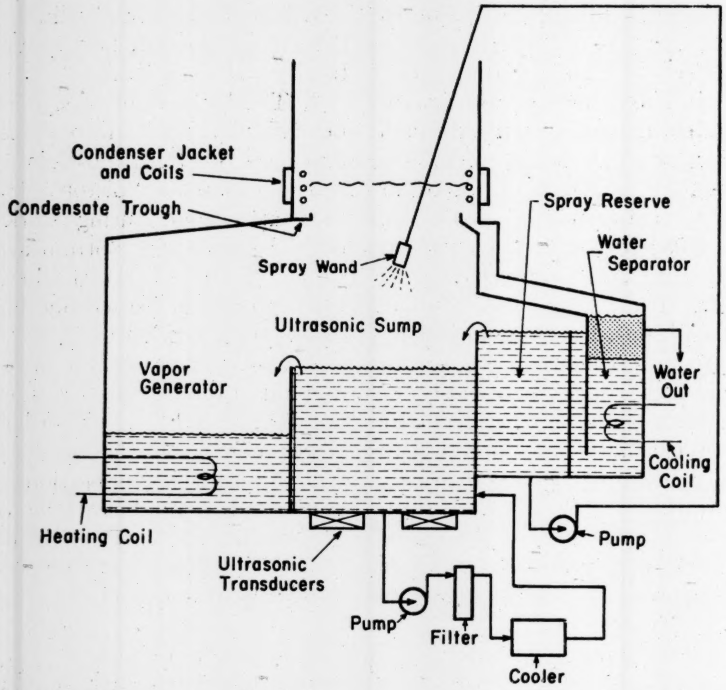
Condenser Jacket and Coils
Condensate Trough
Spray Wand
Spray Reserve
Water Separator
Ultrasonic Sump
Vapor Generator
Water Out
Cooling Coil
Heating Coil
Ultrasonic Transducers
Pump
Pump
Filter
Cooler

FIG. 3.—Trichlorotrifluoroethane Vapor Degreaser with Offset Vapor Generator.

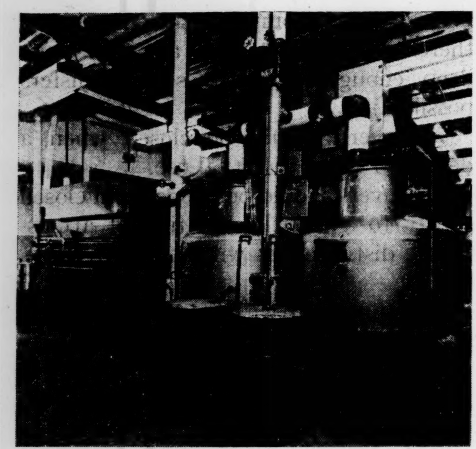

FIG. 4.—Vapor Degreaser Equipped with Adsorbers for Vapor Recovery.

air diffusional losses. Among these are the employment of an offset vapor generating sump (see Fig. 3) to minimize the vapor-air interfacial area available for diffusion, the use of as deep a freeboard as possible to increase the diffusional distance and to minimize the effects of drafts and other sources of air movement around the degreaser, and, finally, the use of carbon adsorbers (Fig. 4) on large machines to recover the solvent losses.

Where it can be used, an offset generator should reduce the vapor-air diffusional losses by approximately 50 per cent over those of a conventional fully open-top machine.

The diffusional losses from a degreaser theoretically are inversely proportional to the freeboard height. Therefore, to minimize such losses the freeboard height on a machine should be as deep as possible within practical limits. In this regard, trichloroethylene and perchloroethylene degreasers are usually constructed with a freeboard depth equivalent to 50 to 60 per cent of the working width of the machine, while most of the trichlorotrifluoroethane machines currently being placed in service possess freeboard depths on the order of 80 to 100 per cent of the working width of the machine.

As evidenced by the data of Table 8, adsorption on activated carbon is a highly efficient means of recovering trichlorotrifluoroethane from solvent vapor-air mixtures. Recovery equipment (Fig. 4) employing this technique has been incorporated into some large degreaser installations. In these instances, it appears that the adsorbers are effecting a recovery of about 65 to 70 per cent of the solvent that disappears from the degreasers. The remaining 30 to 35 per cent is either recovered in the distillation of the sump heels or lost due to leaks, handling spills, dragout, or incomplete capture of the solvent vapors by the vapor collection system.

TABLE 8.—RECOVERY OF TRICHLOROTRIFLUOROETHANE BY CARBON ADSORPTION—TYPICAL LABORATORY TEST RESULTS.

Tests made with a carbon adsorber 36 in. in diameter with a bed depth of approximately 18 in. Bed contained 300 lb of 4 by 10 mesh activated carbon. Superficial gas velocity in bed was approximately 100 ft per min. Trichlorotrifluoroethane vaporized and mixed with adsorber inlet air to yield solvent-air feed mixtures of various concentrations.

Inlet Solvent in Air Concentration, ppm by volume	Bed Loading During Test, lb solvent introduced per 100 lb carbon	Per Cent Solvent Recovery
91	4.0	100
186	3.7	99
326	10.4	95
353	20.7	91
988	10.3	96
953	20.7	95
985	31.1	94

In degreasers operating with trichlorotrifluoroethane, the diffusional losses arising from the presence of water vapors are not the problem that they can be in trichloroethylene or perchloroethylene units because of the solvent-water equilibria previously discussed under the subject of moisture control. As noted therein, the reduction in vapor density occasioned by the co-distillation of solvent and water is much less severe in trichlorotrifluoroethane machines than it is in trichloroethylene or perchloroethylene degreasers (9 per cent versus 30 to 57 per cent).

Solvent vapor losses also may result from a failure to maintain flow in the condenser water system. It is good practice to install a safety thermostat in the freeboard zone, just above the normal vapor line, to shut off the heat to the unit if the vapors rise above the condenser. This thermostat generally is set to turn off the heat when the gas temperature in its vicinity reaches 100 to 105 F. As noted above, this thermostat is strictly a safety device and it should not be regarded as an operating control. If so used, vapor losses will be excessive because of the intermittent escape of solvent vapors past the condenser.

The drag-out losses will be a function primarily of the work being processed and work basket design. Careful attention should be given to the design of work baskets and to the methods of racking the work for cleaning so as to ensure that a minimum of solvent will be trapped in the work or the baskets. The baskets should not be any heavier or larger than absolutely necessary and should be constructed to provide for free drainage of solvent back into the machine.

Drag-out losses also can be minimized by allowing the work to remain in the vapor zone until all condensation has ceased. Low-temperature vapor degreasing generally is advantageous in this respect because of the shorter time required to heat the work up to the vapor temperature. For this reason, drag-out losses resulting from operator impatience are not as likely to be encountered with trichlorotrifluoroethane machines as with units employing the higher boiling chlorinated solvents. Drainage of liquid solvent from the work also is facilitated by the low surface tension possessed by trichlorotrifluoroethane.

Losses due to leaks and spills and to improper use of the degreaser can be minimized and prevented, of course, by careful supervision and proper maintenance of the degreaser.

The boiling sump of any degreaser has to be cleaned out periodically to dispose of the accumulated soils. The dirty solvent drained from the degreaser can be completely reclaimed if proper distillation equipment and procedures are used. The solvent drained from the machine either should be drummed and subsequently charged to a batch still, or pumped directly from the degreasing unit to the still system. The latter, of course, eliminates the solvent handling operations with their attendant losses. On large units or on units where it is desired to maintain continuously a minimum level of contamination in the

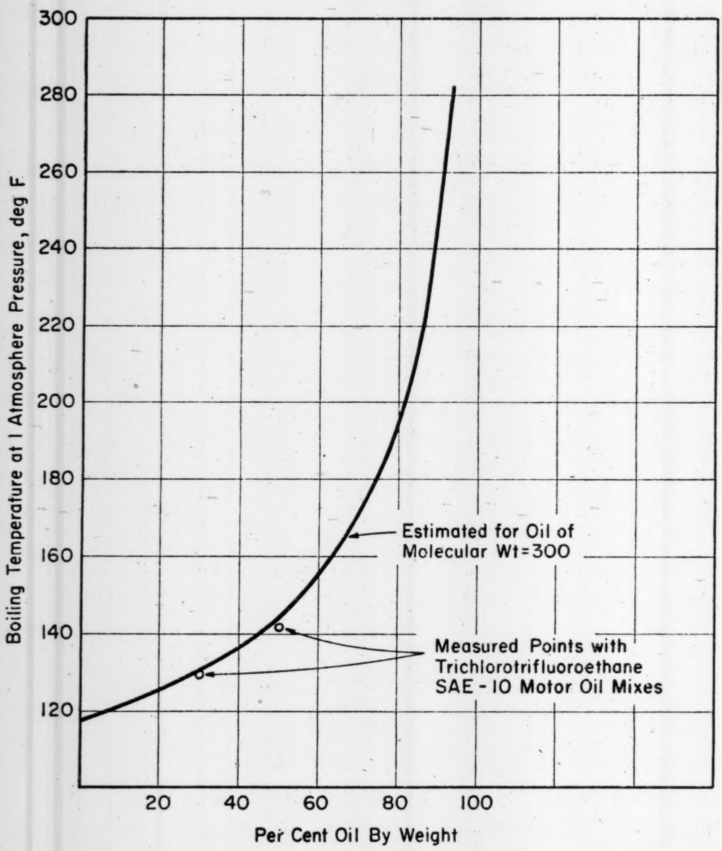

FIG. 5.—Boiling Point of Trichlorotrifluoroethane - Mineral Oil Mixtures.

degreaser, the solvent in the boiling sump is usually pumped in a semicontinuous fashion to a constant level still.

It is readily apparent from Fig. 5 that the bulk of the solvent can be reclaimed at temperatures easily attainable with low-pressure steam heating. However, to attain maximum recovery of solvent, provisions should be made for the injection of live steam into the bottom of the still pot to steam strip the solvent from the still heel. It is generally advisable to begin the live steam injection when the still temperature approaches 170 to 180 F.

The cleanout cycle will be determined by the nature of the object being cleaned, the volume of work, and the amount and type of soil being removed. Sometimes the cleanout cycle will be dictated by the rate of accumulation of insolubles in the degreaser. However, with multicompartment units, the operating life of a solvent charge can usually be extended by continuously filtering the solvent in the wash compartments to free it of insoluble matter. More often, the cleanout cycle is dictated by the accumulation of oil dissolved in the solvent in the boiling sump of the machine. As the solvent's oil content increases, the boiling point of the mixture increases (Fig. 5). This rise in solvent boiling point or the change in the liquid

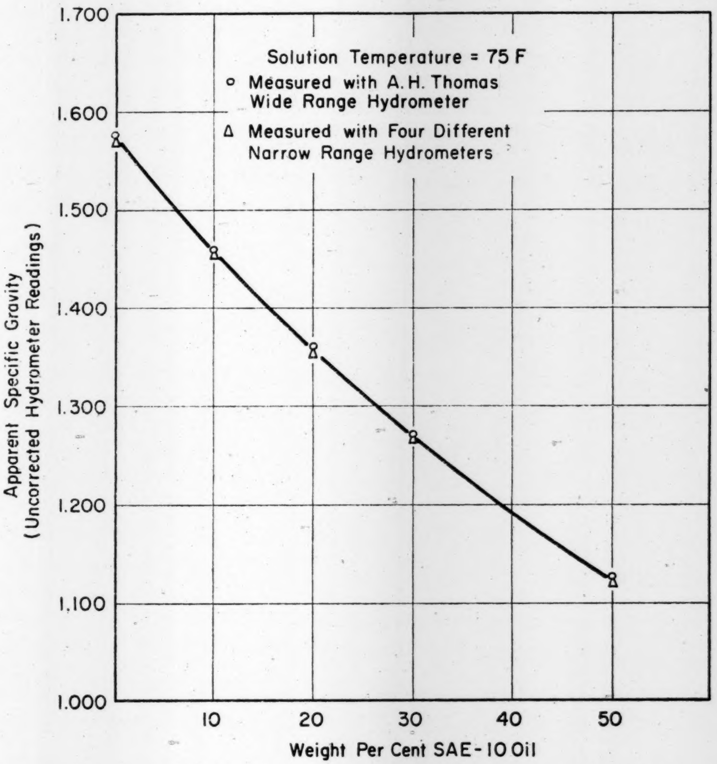

FIG. 6.—Weight Per Cent SAE-10 Oil in Oil-Trichlorotrifluoroethane Mixtures at Various Hydrometer Readings.

density of the solvent in the boiling sump (Fig. 6) are often used as guides for determining when a cleanout is necessary. Sometimes a thermostat is placed in the boiling sump as a safety measure to turn off the heat in the event that the sump contamination for one reason or another exceeds the maximum permissible level. It is generally considered good practice to limit the oil contamination of the solvent in the boiling sump to a maximum value of about 25 to 30 per cent.

SUMMARY

In summary, this article has reviewed the physical and chemical properties of trichlorotrifluoroethane. Basic considerations involved in the design, installation, and operation of vapor degreasing equipment for the utilization of this versatile solvent also have been reviewed.

APPENDIX I

A FIELD METHOD OF DETECTING ALCOHOLS IN TRICHLOROTRIFLUOROETHANE SOLVENT

Ferrox test papers provide a simple field method of detecting small concentrations (down to 0.3 weight per cent) of alcohols in trichlorotrifluoroethane solvent. In the presence of alcohols, these test papers release a purple dye which colors the solvent being tested. In the absence of alcohols,[7] no color development takes place in the solvent.

The Ferrox test papers are prepared by dipping laboratory filter paper (Whatman No. 50) in a solution consisting of 1.62 g of ferric chloride and 2.28 g of ammonium thiocyanate in just enough distilled water to solubilize these compounds. After dipping in the foregoing solution, the papers are hung on a line to air dry. After drying, the papers are cut into $\frac{1}{4}$ in. wide strips and stored in a brown bottle.[8]

In testing for the presence of alcohols, a strip of the Ferrox test paper is placed in a test tube, bottle, or vial containing the solvent under test. If a color bleed into the solvent is not experienced within 15 min, it can be concluded that the solvent contains less than 0.3 weight per cent of alcohol contamination.

APPENDIX II

ESTIMATION OF MAXIMUM HEAT FLUX FOR NUCLEATE BOILING OF TRICHLOROTRIFLUOROETHANE SOLVENT

Per Kutateladse's correlation (**4**), peak heat flux for nucleate boiling at atmospheric pressure is given by the following expression:

$$\frac{q}{A} = \frac{\pi}{24} h_{fg}(gp_v)^{1/2}\,[\sigma(p_l - p_v)]^{1/4}$$

where:

h_{fg} = latent heat of vaporization = 63.14 Btu per lb,

g = acceleration due to gravity = 4.17×10^8 ft per hr^2,

p_v = vapor density = 0.4597 lb per cu ft,

p_l = liquid density = 94.29 lb per cu ft,

σ = surface tension = 1.125×10^{-3} lb per ft, and

$\frac{q}{A}$ = peak heat flux, Btu per hr sq ft.

$$\therefore \frac{q}{A} = \frac{\pi}{24}(63.14)\,[(4.17 \times 10^8)\,(0.4597)]^{1/2}$$

$$[(1.125 \times 10^{-3})\,(94.29 - 0.46)]^{1/4}$$

$$= (8.26)\,(1.386 \times 10^4)\,(5.701 \times 10^{-1})$$

$$= 65{,}300 \text{ Btu per hr sq ft}$$

---

[7] Color development *will also occur* if the solvent is contaminated with ketones, methyl cellosolve, ethers, acetates, organic phosphates, or gross quantities of water. This test *does not detect* contamination with either hydrocarbon or other halogenated solvents. Alkaline substances *will interfere* with the detection of alcohols by the Ferrox test papers since they will bleach the ferric thiocyanate dye.

[8] Ferrox test papers degrade with prolonged exposure to light.

APPENDIX III

ESTIMATION OF DEGREASER HEAT LOSSES DUE TO RADIATION AND CONVECTION

According to *Chemical Engineers' Handbook* (9), combined heat loss due to radiation and convection is given by the following expression:

$$Qrc = (hc + hr)\,(A)\,(\Delta t)$$

where:

Qrc = combined heat loss due to radiation and convection, Btu per hr,

hc = coefficient for convective heat transfer, Btu per hr sq ft deg F, = 0.27 $(\Delta t)^{0.25}$,

Δt = temperature difference, wall to room ambient, deg F,

hr = coefficient for radiation heat transfer, Btu per hr sq ft deg F,

$$= \frac{\left[\left(\frac{T_1}{100}\right)^4 - \left(\frac{T_2}{100}\right)^4\right](0.173)(\epsilon)}{T_1 - T_2}$$

T_1 = wall temperature, deg R,

T_2 = ambient temperature, deg R,

ϵ = emissivity, dimensionless, = 0.66 for chromium-nickel alloy, and

A = wall area, sq ft.

Assume room ambient temperature to be 75 F. Then, with trichlorotrifluoroethane degreaser,

Δt = 117.6 − 75 = 42.6 F,

T_1 = 117.6 + 459.6 = 577.2 R,

T_2 = 75 + 459.6 = 534.6 R,

hc = $(0.27)\,(42.5)^{0.25} = (0.27)\,(2.555) = 0.69$,

$$hr = \frac{\left(\frac{577.2}{100}\right)^4 - \left(\frac{534.6}{100}\right)^4}{577.2 - 534.6}\,(0.173)\,(0.66)$$

$$= \frac{1110 - 816}{42.6}\,(0.173)\,(0.66)$$

$$= (6.91)\,(0.173)\,(0.66) = 0.79, \text{ and}$$

$$\frac{Qrc}{A} = (0.69 + 0.79)\,(42.6) = 1.48\,(42.6)$$

$$= 63 \text{ Btu per hr sq ft}$$

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288-A	Bibliography and Abstracts on Thermostat Metal 1960–1961
315-A	Engine Test Sequences for Evaluating Automotive Lubricants for API Service MS
320	Symposium on Masonry Testing
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327	Symposium on Standards for Filament-Wound Reinforced Plastics
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337	Papers on Industrial Water and Industrial Waste Water
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